

SECTION III

SAMPLE CONTAINER PREPARATION AND CLEANING PROCEDURES

This Section is provided as guidance for the preparation of sample containers that meet the contaminant-free specifications contained in Section II. There are various procedures for cleaning sample containers depending upon the analyses to be performed on the sample. The following cleaning procedures are modeled after those specified for the Superfund Sample Container Repository program.

- A. Cleaning Procedure for Container Types: A, E, F, G, H, J, and K
 - 1. Sample Type: Semivolatile Organics, Pesticides, Metals, Cyanide, and Fluoride in Soils and Water.
 - a. Wash glass bottles, teflon liners, and caps with hot tap water using laboratory grade nonphosphate detergent.
 - b. Rinse three times with copious amounts of tap water to remove detergent.
 - c. Rinse with 1:1 nitric acid (reagent grade HNO_3 , diluted with ASTM Type I deionized water).
 - d. Rinse three times with ASTM Type I organic free water.
 - e. Oven dry bottles, liners and caps at 105-125°C for one hour.
 - f. Allow bottles, liners, and caps to cool to room temperature in an enclosed contaminant-free environment.
 - g. Rinse bottles with pesticide grade hexane (for pesticide determinations) or pesticide grade methylene chloride (for semivolatile organics determinations) using 20 mL for 1/2 gallon containers; 10 mL for 32-oz and 16-oz containers; and 5 mL for 8-oz and 4-oz containers.
 - h. Oven dry bottles, liners, and caps at 105-125°C for one hour.
 - i. Allow bottles, liners, and caps to cool to room temperature in an enclosed contaminant-free environment.
 - j. Place liners in lids and cap containers.
 - k. Label each container with lot number and pack in case.
 - l. Label exterior of each case with lot number.
 - m. Store in contaminant-free area.

2. Sample Type: Nitrate/Nitrite in Soils and Water.
 - a. Substitute reagent grade sulfuric acid (H_2SO_4) for nitric acid in step A.1.c.
 - b. Follow all other steps in the cleaning procedure described in part A.1 above.
- B. Cleaning Procedure for Container Types: B, D
 1. Sample Type: Purgeable (Volatile) Organics in Soils and Water.
 - a. Wash glass vials, teflon-backed septa, teflon liners, and caps in hot water using laboratory grade nonphosphate detergent.
 - b. Rinse three times with copious amounts of tap water to remove detergent.
 - c. Rinse three times with ASTM Type I organic-free water.
 - d. Oven dry vials, caps, septa, and liners at 105-125°C for one hour.
 - e. Allow vials, caps, septa and liners to cool to room temperature in an enclosed contaminant-free environment.
 - f. Seal 40-mL vials with septa (teflon side down) and cap.
 - g. Place liners in lids and cap 120-mL vials.
 - h. Label each vial with lot number and pack in case.
 - i. Label exterior of each case with lot number.
 - j. Store in contaminant-free area.
- C. Cleaning Procedure for Container Types: C, L
 1. Sample Type: Metals, Cyanide, and Fluoride in Soils and Water.
 - a. Wash polyethylene bottles and caps in hot tap water using laboratory-grade nonphosphate detergent.
 - b. Rinse three times with copious amounts of tap water to remove detergent.
 - c. Rinse with 1:1 nitric acid (reagent grade HNO_3 , diluted with ASTM Type I deionized water).
 - d. Rinse three times with ASTM Type I deionized water.

- e. Invert and air dry in contaminant-free environment.
 - f. Cap bottles.
 - g. Label each container with lot number and pack in case.
 - h. Label exterior of each case with lot number.
 - i. Store in contaminant-free area.
2. Sample Type: Nitrate/Nitrite in Soils and Water.
- a. Substitute reagent grade sulfuric acid (H_2SO_4) for nitric acid in step C.1.c.
 - b. Follow all other steps in the cleaning procedure described in part C.1 above.

SECTION IV

SAMPLE CONTAINER QUALITY ASSURANCE AND QUALITY CONTROL REQUIREMENTS

A. Quality Assurance

The objectives of this Section are to: (1) present procedures for evaluating quality assurance (QA) information to ensure that specifications identified in Section II have been met; and (2) discuss techniques for the quality control (QC) analysis of sample containers to be used in conjunction with the cleaning procedures contained in Section III.

Major QA/QC activities should include the inspection of all incoming materials, QC analysis of cleaned lots of containers, and monitoring of the containers' storage area. Complete documentation of all QC inspection results (acknowledging acceptance or rejection) should be kept as part of the permanent bottle preparation files. QA/QC records (e.g., preparation/QC logs, analytical data, data tapes, storage log) should also be stored in a central location within the facility.

Documentation indicating that the container lot has passed all QA/QC requirements should be provided by the bottle vendor to the bottle purchaser with each container lot. Documentation should include a signed and dated cover statement affirming that all QA/QC criteria were met or exceeded and copies of raw data from applicable analyses of the QC containers. Minimum documentation that should be provided with each lot of containers follows:

- A statement that "Sample container lot _____ meets or exceeds all QA/QC criteria established in 'Specifications and Guidance for Obtaining Contaminant-Free Sample Containers'";
- Reconstructed Ion Chromatographs (RICs) from volatile and semivolatile organics determinations;
- GC chromatographs from pesticides determinations;
- ICP, hydride-ICP, or ICP-MS instrument readouts from metals determinations;
- AA raw data sheets and instrument readouts from metals determinations; and
- Cyanide, fluoride, and nitrate/nitrite raw data sheets and instrument readouts from these determinations.

1. Incoming Materials Inspection:

A representative item from each case of containers should be checked for conformance with specifications provided in Section II. Any deviation should be considered unacceptable. A log of incoming shipments should be maintained to identify material type, purchase order number, and delivery date. The date

of incoming inspection and acceptance or rejection of the material should also be recorded on this log.

2. Quality Control Inspection of Cleaned Lots of Containers:

Following container cleaning and labeling, two containers should be selected from each container lot to be used for QC purposes. The two categories of QC containers should be as follows:

a. Analysis QC Containers:

One QC container per lot should be designated as the analysis QC container. The sample container preparer should analyze the analysis QC container(s) to check for contamination prior to releasing the associated container lot for shipment. The QC analyses procedures specified in the Quality Control Analysis part of this Section for determining the presence of semivolatile and volatile organics, pesticides, metals, cyanide, fluoride, and nitrate/nitrite should be utilized.

For each representative analysis QC container(s), the appropriate QC number should be assigned to the related lot of containers. For example, the QC number could be a six-digit number sequentially assigned to each lot that has undergone QC analysis. Under this numbering scheme, the first alphabetical character would be the container type letter from Figure 1, the next four digits would be assigned sequentially in numerical order starting with "0001" for the first lot to undergo QC analyses, and the last character would be either a "C" to indicate clearance or an "R" to indicate rejection.

If the representative analysis QC container(s) passes QC inspection, the related lot of containers should be released, and the appropriate QC number should be entered in the preparation/QC log to indicate clearance of the lot for shipment.

If the analysis QC container(s) are found to be contaminated per the specified QC analysis procedures, the appropriate QC rejection number should be assigned and entered in the preparation/QC log. Any container labels should be removed and the entire lot returned for reprocessing under a new lot number. Excessive QC rejection for a particular container type should be noted for future reference.

A laboratory standard and a blank should be run with each QC analysis. All QC analysis results should be kept in chronological order by QC report number in a central QC file. The QC numbers assigned should be documented in the preparation/QC log, indicating acceptance or rejection and date of analysis.

A container lot should not be released for shipment prior to QC analysis and clearance. Once the containers have passed QC inspection, the containers should be stored in a contaminant-free area until packaging and shipment.

b. Storage QC Containers:

One QC container per lot should be designated as the storage QC container. The storage QC container should be separated from the lot after cleaning and labeling and should be stored in a designated contaminant-free area for one year. The date the container is placed in the storage area should be recorded in the storage QC container log.

If contamination of the particular container lot comes into question at any time following shipment, the storage QC container should be removed from the storage area and analyzed using the QC analysis procedures for that container type (see Quality Control Analysis, this Section). Upon removal, containers should be logged out of the storage area.

The designated storage area should be monitored continuously for volatile contaminants. A precleaned, 40-mL vial that has passed a QC inspection should be filled with ASTM Type I organic-free water and be placed in the storage area. This vial should be changed at one-week intervals. The removed vial should be subjected to analysis for volatile organics as described in the Quality Control Analysis part of this Section. Any peaks indicate contamination. Identify contaminants, if present, and include the results in a report to all clients who purchased bottles in the past month from the affected lot(s).

B. Quality Control Analysis

The types of QC analyses correlate with the types of containers being analyzed and their future use in sample collection. The QC analyses are intended for the determination of:

- Semivolatile organics and pesticides;
- Volatile organics;
- Metals;
- Cyanide;
- Fluoride; and
- Nitrate/Nitrite.

QC analyses should be performed according to the container type and related sample type and utilize the specific method(s) described below.

1. Determination of Semivolatile Organics and Pesticides:

Container Types: A, E, F, G, H, J, and K

a. Sample Preparation:

- Add 60 mL of pesticide-grade methylene chloride to the container, and shake for two minutes.
- Transfer the solvent to a Kuderna-Danish (KD) apparatus equipped with a three-ball Snyder column. Concentrate to less than 10 mL on a steam bath. Split the solvent into two 5 mL fractions for semivolatile and pesticide determinations.
- Add 50 mL of pesticide-grade hexane (for pesticide determinations only) to the KD apparatus by slowly pouring down through the Snyder column. Concentrate to less than 10 mL to effect solvent replacement of hexane for methylene chloride.
- Concentrate the solvent to 1 mL using a micro-Snyder column.
- Prepare a solvent blank by adding 60 mL of the rinse solvent used in step "g" of the cleaning procedure for container types A, E, F, G, H, J, and K (Section III page 14) directly to a KD apparatus, and proceed as above.

b. Semivolatile Organics Sample Analysis:

- Instrument calibration should be performed as described in the current CLP Low Concentration Organics SOW with the following exceptions:
 - (1) If problems are encountered meeting the \pm RSR criteria on the initial calibration for semivolatiles, the high concentration point should be deleted and a four-point calibration used.
 - (2) The low concentration standard should be used for the continuing calibration standard for semivolatile analyses.
 - (3) The percent difference window should be widened to \pm 30 percent for all compounds.
- Inject 1 μ L of solvent into a gas chromatograph/mass spectrometer (GC/MS).
- GC/MS operating conditions are listed in Figure 3 (page 28).
- Any peaks found in the container solvent that are not found in the solvent blank or with peak heights or areas not within \pm 50 percent of the blank peak height or area should be cause for rejection.

- Identify and quantitate any contaminant(s) that cause rejection of a container lot.
 - A standard mixture of the 9 semivolatile organic compounds listed in Table 3 (page 27) with concentrations in the 5-20 ppb range should be analyzed to ensure that sensitivities are achieved that will meet contract required quantitation limits.
 - A solvent blank should be run with each analysis.
- c. Pesticides Sample Analysis:
- Instrument calibration should be performed as described in the current CLP Low Concentration Organics SOW.
 - Inject 1 μ L of solvent into a gas chromatograph (GC) equipped with an electron capture detector (ECD).
 - GC/ECD operating conditions are listed in Figure 4 (page 29).
 - Any peaks found in the container solvent that are not found in the solvent blank or with peak heights or areas not within + 50 percent of the blank peak height or area should be cause for rejection.
 - A standard mixture of the 7 pesticide compounds listed in Table 3 (page 27) with concentrations in the 0.01 to 1 ppb range should be analyzed to ensure that sensitivities are achieved that will meet contract required quantitation limits.
 - A solvent blank should be run with each analysis.

2. Determination of Volatile Organics:

Container Types: B and D

a. Sample Preparation:

- Fill the container with ASTM Type I organic-free water.

b. Sample Analysis:

- Instrument calibration should be performed as described in the current CLP Low Concentration Organics SOW with the following exceptions:
 - (1) If problems are encountered meeting the \pm RSR criteria on the initial calibration for volatiles, the high concentration point should be deleted and a four-point calibration used.

- (2) The low concentration standard should be used for the continuing calibration standard for volatile analyses.
- (3) The percent difference window should be widened to ± 30 percent.
- GC/MS operating conditions are listed in Figure 5 (page 30).
- Any peaks not found in the blank or with peak heights or areas not within ± 50 percent of the blank peak height or area should be cause for rejection.
- Identify and quantitate any contaminant(s) that cause rejection of a container lot.
- A standard mixture of the 5 volatile organic compounds listed in Table 3 (page 27) with concentrations in the 1-5 ppb range should be analyzed to ensure that sensitivities are achieved that will meet contract required quantitation limits.
- A blank should be run by analyzing an aliquot of the ASTM Type I water used above.

3. Determination of Metals:

Container Types: A, C, E, F, G, H, J, K and L

a. Sample Preparation:

- Add 50 mL of ASTM Type I deionized water to the container, and acidify with 0.5 mL reagent-grade HNO_3 . Cap and shake well.
- Treat the sample as a dissolved metals sample. Analyze the undigested water using the current CLP Low Concentration Inorganics SOW.

b. Sample Analysis:

- Instruments used for the analysis of the samples should meet the contract required detection limits in Table 1.
- The rinse solution should be analyzed before use on the bottles that are designated for analysis to ensure that a contaminated solution is not used for rinsing the bottles.
- Calibration verification standards should be analyzed at the beginning, end, and every ten samples within an analysis run (a continuous analytical sequence consisting of prepared samples and all associated quality assurance measurements). The verification standards should be three to five times the values in Table 1. The percent recovery factor for the

verification standards should be between 90 to 110 percent or ± 10 percent of the actual value of the verification standard.

- Calibration blanks should be analyzed at the beginning, end, and every ten samples within an analysis run. A calibration blank is a solution made up exactly like the sample preparation solution. The calibration blank should be below the values in Table 1.
- A set of standards in the expected working range should be analyzed with each analytical run. The acid matrix of the standards, blank, and quality control samples should match that of the samples.
- Concentrations at or above the detection limit for each parameter (listed in Table 1) should be cause for rejection of the lot of containers. NOTE: Sodium detection limit for container types A, E, F, G, H, J, and K is 5000 $\mu\text{g/L}$ unless the containers will be used for low concentration analyses, then the detection limit is 500 $\mu\text{g/L}$.

4. Determination of Cyanide:

Container Types: A, C, E, F, G, H, J, K and L

a. Sample Preparation:

- Place 250 mL of ASTM Type I deionized water in the container. Add 1.25 mL of 6N NaOH (for container types F and G use 100 mL ASTM Type I deionized water and 0.5 mL 6N NaOH). Cap the container and shake vigorously for two minutes.

b. Sample Analysis:

- Analyze an aliquot as described in the current CLP Low Concentration Inorganics SOW.
- The detection limit should be 10 $\mu\text{g/L}$ or lower.
- A blank should be run by analyzing an aliquot of the ASTM Type I water used above.
- A set of standards in the expected working range, a quality control sample, and blank should be prepared exactly as the sample.
- The detection of contaminants of 10 $\mu\text{g/L}$ cyanide (or greater) should be cause for rejection of the lot of containers. NOTE: Contamination could be due to the container, the cap, or the NaOH.

5. Determination of Fluoride:

Container Types: A, C, E, F, G, H, J, K and L

a. Sample Preparation:

- Place 250 mL of ASTM Type I deionized water in the container (for container types F and G use 100 mL ASTM Type I deionized water). Cap the container and shake vigorously for two minutes.

b. Sample Analysis:

- Analyze an aliquot as described in the current CLP Low Concentration Inorganics SOW.
- The detection limit should be 200 $\mu\text{g/L}$ or lower.
- A blank should be run by analyzing an aliquot of the ASTM Type I water used above.
- A set of standards in the expected working range, a quality control sample, and blank should be prepared exactly as the sample.
- The detection of contaminants of 200 $\mu\text{g/L}$ (or greater) fluoride should be cause for rejection of the lot of containers. NOTE: Contamination could be due to the container or the cap.

6. Determination of Nitrate/Nitrite:

Container Types: A, C, E, F, G, H, J, K and L

a. Sample Preparation:

- Place 250 mL of ASTM Type I deionized water in the container (for container types F and G use 100 mL ASTM Type I deionized water). Cap the container and shake vigorously for two minutes.

b. Sample Analysis:

- Analyze an aliquot as described in the current CLP Low Concentration Inorganics SOW.
- The detection limit should be 100 $\mu\text{g/L}$ or lower.
- A blank should be run by analyzing an aliquot of the ASTM Type I water used above.

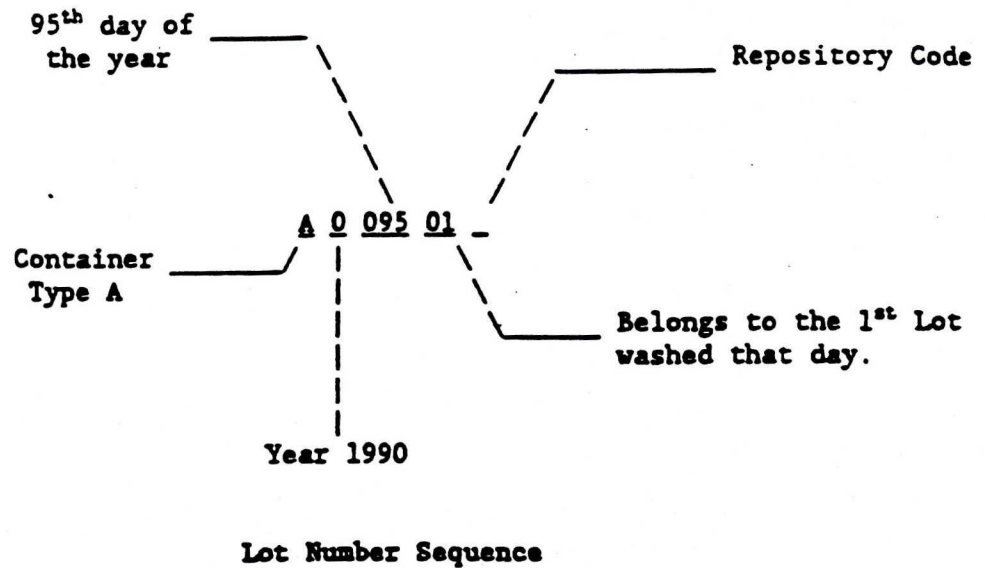
- A set of standards in the expected working range, a quality control sample, and blank should be prepared exactly as the sample.
- The detection of contaminants of 100 $\mu\text{g/L}$ (or greater) nitrate/nitrite should be cause for rejection of the lot of containers. NOTE: Contamination could be due to the container or the cap.

C. Preparation and Labeling

Sampling for environmental specimens requires that sample containers be transported to field sites prior to sample collection. As a result, considerable time may elapse between the receipt of sample containers and collection of the samples. Because of the large number of samples taken at any one site, accounting for all sample containers can become extremely difficult. The following guidance on the identification and tracking of sample containers is based on procedures that have been used successfully in the CLP bottle program.

1. Each shipment should be inspected to verify that the requested number of cleaned and prepared sample containers have been supplied and meet the requirements specified in Section II (Tables 1 and 2). If any shipment fails to meet the required specifications, it should be discarded and replaced with a supply of sample containers that meet the required criteria.
2. The sample containers should be removed and prepared in accordance with the methods designated below:
 - a. Allocate the appropriate number of sample containers (Figure 1) to a designed container lot.
 - b. Recommended lot size for each container should be based on the recommended number of items per case.
3. A permanent eight-digit lot number should be assigned to each lot of sample containers for identification and tracking purposes throughout the life of the containers. Figure 2 provides an example of a lot number sequence.

FIGURE 2



- a. The first digit represents the container type in Section II (Figure 1).
 - b. The second digit represents the last digit of the calendar year.
 - c. The next three digits represents the day of the year on which the sample containers were washed.
 - d. The sixth and seventh digits represent the daily lot number.
 - e. The final digit represents the identification of the person who prepared the lot.
4. The lot number for each container should be entered, along with the date of washing, type of container, and number of containers per lot, into the preparation/QC log book.
 5. Lot numbers printed with solvent resistant ink on a nonremovable label should remain with the corresponding containers throughout the cleaning procedure.
 6. After sample container cleaning and drying, the label should be affixed to the containers in a permanent manner.
 7. At least one face should be clearly marked, excluding the top and bottom faces, of each case of sample containers with the assigned lot numbers.

TABLE 3

STANDARD MIXTURES OF ORGANIC COMPOUNDS TO VERIFY SENSITIVITY

<u>Volatiles</u>	<u>Semivolatiles</u>	<u>Pesticides</u>
Methylene Chloride	Nitrobenzene	Gamma-BHC
Acetone	4-Chloroaniline	Heptachlor
2-Butanone	2,6-Dinitrotoluene	Aldrin
Trichloroethene	Diethylphthalate	Dieldrin
Toluene	4-Bromophenyl-phenylether	Endrin
	Hexachlorobenzene	4,4'-DDT
	Pentachlorophenol	Aroclor 1260
	Di-n-butylphthalate	
	bis(2-Ethylhexyl)phthalate	

FIGURE 3

GC/MS OPERATING CONDITIONS FOR SEMIVOLATILE ORGANICS QC ANALYSIS

OPERATOR: _____	DATE: _____
JOB NUMBER: _____	SAMPLE IDENTIFICATION: <u>Container Lot number</u>
SOLVENT: <u>Methylene Chloride</u>	ANALYTICAL METHOD: <u>CLP Low Concentration SOW</u>
<u>Semivolatile Organics Fraction</u>	
COLUMN	FID GLASS
Type <u>Fused Silica Capillary or equiv.</u>	Hydrogen, mL/min <u>N/A</u>
Length <u>30 m</u>	Air, mL/min _____
Diameter <u>0.25 mm or 0.32 mm ID</u>	
Liquid Phase (% wt) <u>J&W Scientific DB-5 or equivalent</u>	CHART SPEED, cm/min _____
Support <u>N/A</u>	DETECTOR <u>Mass Spectrometer</u>
Mesh <u>N/A</u>	Range <u>35-500 a.m.u.</u>
	Attenuation _____
CARRIER GAS <u>Helium</u>	
Rotameter _____	TEMPERATURE, °C
Inlet Pressure, psig _____	Detector _____
Linear Velocity cm/sec <u>25-30</u>	Injection Port <u>250-230°C</u>
	Column
SCAVENGER GAS _____	Initial <u>40°/3 min</u>
	Program <u>10°/min</u>
SPLIT _____	Final <u>290°C</u>
	INSTRUMENT _____

FIGURE 4

GC/ECD OPERATING CONDITIONS FOR PESTICIDES QC ANALYSIS

OPERATOR: _____	DATE: _____
JOB NUMBER: _____	SAMPLE IDENTIFICATION: <u>Container Lot number</u>
SOLVENT: <u>Hexane</u>	ANALYTICAL METHOD: <u>CLP Low Concentration SOW</u>
<u>Pesticide Fraction</u>	
COLUMNS (Two are required)	FID GLASS
Type <u>Fused Silica Capillary or equiv.</u>	Hydrogen, mL/min <u>N/A</u>
Length <u>30 m</u>	Air, mL/min <u>N/A</u>
Diameter <u>0.53 mm ID</u>	
Liquid Phase (% wt)	CHART SPEED, cm/min <u>1 cm/min</u>
<u>J&W Scientific DB-1710 and DB-608 or equivalent</u>	
Support <u>N/A</u>	DETECTOR <u>Electron Capture</u>
Mesh <u>N/A</u>	Range _____
	Attenuation <u>16</u>
CARRIER GAS <u>Helium or Hydrogen</u>	
Rotameter _____	TEMPERATURE, °C
Inlet Pressure, psig _____	Detector <u>350°C</u>
Flow Rate, mL/min <u>5</u>	Injection Port <u>≥ 200°C</u>
	Column
SCAVENGER GAS _____	Initial <u>150°/30sec</u>
	Program <u>5-6°/min</u>
SPLIT _____	Final <u>275°C</u>
	INSTRUMENT _____

FIGURE 5

GC/MS OPERATING CONDITIONS FOR VOLATILES QC ANALYSIS

OPERATOR: _____	DATE: _____
JOB NUMBER: _____	SAMPLE IDENTIFICATION: <u>Container Lot number</u>
SOLVENT: <u>Methanol</u>	ANALYTICAL METHOD: <u>CLP Low Concentration SOW</u>
<u>Volatile Organics Fraction</u>	
COLUMN	FID GLASS
Type <u>Fused Silica Capillary or equiv.</u>	Hydrogen, mL/min <u>N/A</u>
Length <u>30 m</u>	Air, mL/min <u>N/A</u>
Diameter <u>0.53 mm ID</u>	
Liquid Phase (% wt)	CHART SPEED, cm/min _____
<u>J&W Scientific DB-624, Suppelco VOCAL or equivalent</u>	
Support <u>N/A</u>	DETECTOR <u>Mass Spectrometer</u>
Mesh <u>N/A</u>	Range <u>35-300 a.m.u.</u>
	Attenuation _____
CARRIER GAS <u>Helium or Nitrogen</u>	
Rotameter _____	TEMPERATURE, °C
Inlet Pressure, psig _____	Detector _____
Flow Rate, mL/min <u>15</u>	Injection Port _____
	Column
SCAVENGER GAS _____	Initial <u>10°/1-5 min</u>
	Program <u>6°/min</u>
SPLIT _____	Final <u>160°C/all cmpds. elute</u>
	INSTRUMENT _____

APPENDIX F

This appendix has been prepared to present Quality Assurance/Quality Control information specific to the Private Well Sampling Plan (PWSP) prepared for Techalloy Company, Inc. (Techalloy) by Roy F. Weston, Inc. (WESTON®). The PWSP was prepared in accordance with the Administrative Order of Consent issued to Techalloy by the United States Environmental Protection Agency (U.S. EPA). The PWSP was submitted to U.S. EPA in May 1993. The plan addresses protocols associated with the sampling of groundwater wells on private properties within the vicinity of the Techalloy facility. The plan is designed to evaluate whether the private wells may potentially be affected by constituents in the groundwater migrating from the Techalloy facility.

Unless otherwise stated in the PWSP or this appendix, all procedures for investigative activities, QA/QC, and report preparation that are contained in the body of this QAPP are applicable to all portions of the PWSP and all related activities. All applicable QAPP protocols are considered part of the PWSP and will be implemented accordingly.

This appendix presents an overview of the scope and objectives of the PWSP, a summary of the private well sampling effort, PWSP data quality objectives, and the laboratory-specific standard operating procedures (SOPs) for the analytical methodologies that will be used to analyze private well water samples.

The PWSP defines the procedures and methodologies to be implemented, in order to qualitatively, quantitatively, and statistically determine the potential influence of the Techalloy groundwater plume on private well receptors. The PWSP identifies all potential receptors (nine private wells) to be sampled within the area potentially affected by the groundwater plume migrating downgradient of the Techalloy facility.

The private wells will be purged and sampled following the procedures outlined in the PWSP. Analysis of the groundwater samples will be performed by WESTON - Gulf Coast Laboratories in University Park, Illinois. All sample handling, documentation, analytical procedures, and data reporting will be consistent with the protocols outlined in this QAPP and the PWSP.

A summary of the sampling effort is presented in Table 1 and a summary of the data quality objectives is presented in Table 2. In addition to the groundwater analyses presented in the PWSP, each private well groundwater sample will also be analyzed for total suspended solids (TSS). The TSS results will aid in the comparison of the constituent concentrations detected in the unfiltered and filtered groundwater samples for metals analysis. The laboratory SOP for TSS is based on the following analytical protocols: Total Suspended Solids - EPA 600/4-79-020 Method 160.2, and Standard Methods, 17th Edition, Method 2540D. Method 8260 will be used for the private well VOC analysis in order to achieve quantitation limits below the U.S. EPA Maximum Contaminant Levels (MCLs). The SOPs for the analytical methods specific to the private well groundwater analyses are presented in this appendix. The SOPs for the analytical methods common to both the RFI Work Plan analyses and the private well analyses are presented in Appendix D of the QAPP.

Table 1
Summary of Private Well Sampling Effort
Techalloy Company, Inc.
Union, Illinois

Sample Matrix	Field Parameters	Laboratory Parameters	Investigative			Field Duplicate			Field Blank			Matrix Spike/Matrix Spike Duplicate ¹			Matrix Total ²
			No.	Freq.	Total	No.	Freq.	Total	No.	Freq.	Total	No.	Freq.	Total	
Private well groundwater	pH, temperature, specific conductance	VOCs	9	1	9	1	1	1	1	1	1	1	1	1	11
		Metals-filtered	9	1	9	1	1	1	1	1	1	---	---	---	11
		Metals-unfiltered	9	1	9	1	1	1	1	1	1	---	---	---	11
		TSS	9	1	9	1	1	1	1	1	1	---	---	---	11

Notes:

¹ MS/MSDs are not additional samples, but are instead investigative samples on which MS/MSD analyses are performed. MS/MSD analyses are for organic samples only. Duplicate/spike analyses will be performed for inorganic samples.

² The matrix total does not include trip blank samples, MS/MSDs and duplicate/spike samples. One trip blank sample will be shipped with every shipment container of aqueous VOA samples.

Table 2

Data Quality Objectives
Private Well Sampling
Techalloy Company, Inc.
Union, Illinois

Data Quality Objective	Task	Data Collection Activity	Data Type	Data Use	Analytical Level
Potential Receptors	Groundwater Sampling at Private Wells	Field parameter evaluation	pH, conductivity, temperature	SP	I
		Collect and analyze groundwater samples	VOCs	SC, EA	V
			Metals	SC, EA	IV
			TSS	SC, EA	V

Notes and Abbreviations:

Data Use Symbols:

SC - Site Characterization
 EA - Engineering Evaluation
 SP - Sampling Protocol

Analytical Levels:

I - Qualitative Screening with Field Instruments
 II - Field Analysis with Field Instruments
 III - Off-Site Analysis by Analytical Laboratory
 IV - Analyses by Routine Methods
 V - Analyses by Special Methods

RELEASED
2019-007454
July 27, 2020 - TJW**ORGANIC ANALYSIS PROTOCOL**
Gas Chromatography/Mass Spectrometry (GC/MS)
Analysis of Volatile Organic Compounds by Method 8260**CONTROLLED DISTRIBUTION**COPY #: *Uncontrolled*ISSUED TO: *Tech Alloy QAP (VH1)*Full Signature Approvals Are Kept on File
with WESTON®'s Analytics Division
QA Standard Practice Records

REVISION NUMBER: 01

1.0

PURPOSE

To outline the guidelines for the analysis of Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS).

2.0

REFERENCES

This SOP was written using SW-846 Method 8260 as a reference.

3.0

METHOD SUMMARY

This method is used to determine volatile organic compounds in a variety of matrices. It is applicable to water, soil, sediment, sludge and waste drum samples.

This method can be used to quantify most volatile organic compounds that have a boiling point less than 200°F. It is also limited to those compounds that elute as sharp peaks from a capillary column. A listing of applicable compounds and their characteristic ions appears in Table 1.

A portion of sample, measured into a sample vessel, is purged with an inert gas. The volatile compounds are transferred to a trap, containing retarding materials.

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The trap is then backflushed with the inert gas and rapidly heated to effectively transfer the compounds to the GC column. The GC oven is then temperature ramped to separate the compounds and introduce them to the source.

The mass filter separates the ions which are then detected by the analyzer. The data system then provides qualitative and quantitative information concerning the sample.

Those samples suspected of high background levels are screened by GC/FID prior to analysis. Initial dilutions are determined from the screen.

Instrument calibration occurs about every 12 hours, or prior to analysis. Instrument maintenance is performed as needed, on a daily, monthly or yearly basis.

4.0

INTERFERENCES

External interferences can be caused by contaminants from sample containers, preparative glassware and reagents, syringes and columns and manifest themselves as high background and/or discrete peaks. Some contaminants are also introduced through the sample vial seal and/or instrument sample connections. Proper glassware preparation, sample handling and instrument maintenance should eliminate these sources. A laboratory method blank is analyzed prior to any analysis to show absence of any contaminants. Reagent water sampled in the lab and carried through all field operations is also analyzed to show absence of contaminants from field sampling.

Carryover is also another source of contamination. Any time a high level sample is analyzed, the next sample in the batch is checked for carryover. If carryover is suspected, that sample is re-analyzed. The position is rinsed with methanol/water. If carryover is excessive and continues into the next samples, the batch is aborted/paused, the column and trap baked, and/or blanks analyzed until all contamination is absent. If further response is required (i.e. trap replacement) it is documented in the maintenance logbook. See maintenance section.

Internal interferences can be purged from the sample with the target compounds and appear as elevated baselines or distinct peaks. Internal interferences most often manifest themselves as low/high recoveries of surrogate/matrix spike compounds. Matrix interferences vary from sample to sample.

The volatile lab must be free of solvents. Common air-borne lab contaminants, dichloromethane, toluene, 2-butanone and acetone are allowed up to 5X the EQL. Occasionally, higher values for acetone and dichloromethane are allowed, but this is approved before analysis.

4.1 Estimated Quantitation Limits (EQL)

Estimated quantitation limits for each routinely analyzed compound appears in Table 2. EQL's are highly matrix dependent and will vary.

5.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Sample containers, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance and/or specific contract or client requests. Listed below are the holding times and the references that include container and preservation requirements for compliance with the Safe Drinking Water Act (SDWA) and the Clean Water Act (CWA).

Matrix	SW-846
All	14 days (VTS)

VTS = verified time of sampling

All samples received for volatile analysis are refrigerated upon receipt at $4 \pm 2^{\circ}\text{C}$. See specific methods for preservation recommendations or requirements.

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6.0 INSTRUMENTATION AND EQUIPMENT

6.1 GC/MS

A temperature programmable chromatograph interfaced with a mass-selective detector capable of scanning from 35 - 260 amu every second or less using 70 volts of electron energy in the electron ionization mode. The system is capable of producing an acceptable spectrum of bromofluorobenzene when 50 ng is injected on-column.

6.2 Data System

The analytical system is interfaced with a data system capable of continuous acquisition and storage of mass spectral data. The RTE 6/1000 Rev. E and UNIX Series 400 software is capable of plotting specific masses versus time or scan numbers (Extracted Ion Current Profile) and integration of that abundance. The systems also contain the most recent NBS library.

6.3 Current Hardware/Software

- 1 Hewlett-Packard 5995 GC/MS. Equipped with a capillary DB-624 column.
- 1 Hewlett-Packard 5890 GC interfaced with a 5970 MSD. Equipped with a DB-624 capillary column.
- 1 Hewlett-Packard 5890 GC interfaced with a 5971 MSD. Equipped with a stand alone UNIX Series 400 computer system and peripherals. Contains a DB-624 Capillary Column.
- 1 Tekmar LSC 2 in connection with 1 Tekmar ALS 10 station autosamplers for the 5995.
- 1 Tekmar LSC 2000 in connection with an ALS 2050 for the 5890/5970 system.
- 1 OI-Analytical 4460A sample concentrator, MPM-16 Multiple Purging Module (16 stations), MHC-16 Cryo-focusing Module for the 5890/5971 MSD.

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- 1 5890 Hewlett-Packard GC and Tekmar LSC 2 single position sample concentrator equipped with dual FID detectors and connected to a linear chart recorder for sample screening.
- 1 Hewlett-Packard 7974 Magnetic Tape Drive for RTE 6/1000 data archival. Two locked cabinets for tape storage. UNIX system is stand alone.
- RTE 6/1000 Rev. E. and UNIX Series 400 software and peripheral hardware.

6.4 Miscellaneous

- assorted syringes (10, 25, 50, 100, 500 and 1000 uL)
- 5 mL and 25 mL luer-lock gas-tight syringes
- assorted purge vessels (water/soil, 5/25 mL, 5 g)
- top-loading balance, capable of weighing to ± 0.1 g, stainless steel spatula
- assorted amber and clear teflon-lined screw-capped vials (1.5 - 2.0 mL, 3.5 - 5.0 mL)
- cleaned 40 mL vials w/teflon-lined screw-caps

7.0 PREVENTIVE MAINTENANCE

Instrumental maintenance can be categorized as daily, yearly and "as required".

7.1 Daily Maintenance

The most routinely performed maintenance includes:

- position rinse
- tube baking after sample analysis
- oven bake after high level samples

7.2 Yearly Maintenance

The only scheduled yearly maintenance includes changing the pump oil. This is done by an HP representative.

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7.3 "As Required"

Most maintenance is done on an "as needed" basis, is operator determined and can be categorized as GC, Tekmar, or MS related.

7.3.1 GC Related

- change column; condition new column
- clean separator; change separator
- check helium flow rate
- change gas cylinders and moisture trap

7.3.2 MS Related

- clean source/rods and anything associated with that activity

7.3.3 Tekmar Related

- rinse positions
- change positions; change parts of positions
- change transfer line; clean transfer line
- replace trap; condition new trap
- refurbish Tekmar
- check purge pressure and flow rate

Each required maintenance may be performed for a variety of reasons. Certain trouble-flags will indicate what maintenance procedures may be required. Refer to the GC/MS Maintenance Manual. An example appears in Figure 1. The entry number is then transferred to the appropriate operation logbook.

8.0 STANDARDS AND QC SOLUTIONS

- 8.1 All neat standards/kits received are recorded in the Neat Standards Logbook. See Figure 2. A code is written on the bottle/kit and recorded in the log book. All neat standards are then stored in a separate freezer at approximately -10°C until needed. See logbook for examples. The standard is issued a Neat Standards Reference Number which is used to track all standards as they are used as is or in preparation of stock/working solutions.

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8.2 Surrogate Spiking Solution

Stock surrogates are purchased as solutions from Supelco. The following surrogates are used:

Compound	Concentration	Catalog No.
4-Bromofluorobenzene	2000 ug/mL	4-8083
1,2-Dichloroethane-d ₄	2000 ug/mL	4-8941
Toluene-d ₈	2000 ug/mL	4-8087

These are stored in a separate freezer at approximately -10°C in the dark and kept for a period of one year. The working solution is prepared with internal standard solution. See next section.

8.3 Internal Standard Spiking Solutions

Stock internal standards are purchased as solutions from Supelco. The following internal standards are used:

Compound	Concentration	Catalog No.
Pentafluorobenzene	2000 ug/mL	4-8945
Chlorobenzene-d ₅	2000 ug/mL	4-8086
1,4-Difluorobenzene	2000 ug/mL	4-8944
1,2-Dichlorobenzene-d ₄	2000 ug/mL	4-8952

These are stored at -10°C in the dark. Unopened ampules are kept for a period of one year. Opened ampules are kept for a period of 6 months or until used. The contents of the opened ampules is transferred to a 1.5 - 2.0 mL amber teflon-lined screw-capped vial. The WORKING internal standard/surrogate solution is prepared as follows:

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Compound	Volume (uL)	MeOH (mL)	Concentration
Pentafluorobenzene	50	2 mL	50 ppm
Chlorobenzene-d ₅	50		
1,4-Difluorobenzene	50		
1,2-Dichlorobenzene-d ₄	50		
Bromofluorobenzene	50		
1,2-Dichloroethane-d ₄	50		
Toluene-d ₈	50		

This is stored in a 3.5 - 5.0 mL amber teflon-lined screw-capped vial at approximately -10°C in the dark. All standard preparation is recorded in the Volatiles Standard Logbook. See Figure 3. Each standard is issued a Standard Reference Number (SRN). Working ISS/SSS is prepared every two weeks. Addition of 5 uLs to 5 mLs/25 mLs volume results in a concentration of 50/10 ppb (soil/water).

All standard labels contain the following information: standard description, concentration, date prepared, analyst and expiration date. All neat standards are returned to storage.

8.4

Stock Purgeable Standards

These are obtained as neat solutions from Supelco. The contents of each solution, catalog number and concentrations appear on the next page. The ampules are stored at approximately -10°C in the dark. Stock ampules are good for a period of one year. Upon opening, all contents are transferred to 1.5 - 2.0 mL amber, teflon-lined screw-capped vials. Opened stocks can be used for a period of three months. Listed are compounds in Method 8260 and includes those done on a regular basis. Other standards, if needed, are either purchased as neat solutions or neat standards from Supelco, Chem Service or other certified supplier.

See appropriate entries in the Neat Standards/VOA Standards Logbook. All neat standards are still logged into the Neat Standard Logbook.

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 8.4.1 Working 5-Point Standard

Each stock calibration mix is transferred to a 1.5 - 2.0 mL amber teflon-lined screw-capped vial just prior to use. The following tables describes the preparation procedure:

Waters			
Stock Compound/Mix	Volume (uL)	Vol (MeOH)	Conc.
2000 ppm VOC Mix 1	25	diluted to 2 mLs	25 ng/uL each component
2000 ppm VOC Mix 2	25		
2000 ppm VOC Mix 3	25		
2000 ppm VOC Mix 4	25		
2000 ppm VOC Mix 5	25		
2000 ppm VOC Mix 6	25		

Soils			
Stock Compound/Mix	Volume (uL)	Vol (MeOH)	Conc.
2000 ppm VOC Mix 1	100	diluted to 2 mLs	100 ng/uL each component
2000 ppm VOC Mix 2	100		
2000 ppm VOC Mix 3	100		
2000 ppm VOC Mix 4	100		
2000 ppm VOC Mix 5	100		
2000 ppm VOC Mix 6	100		

NOTE: The other standards used in the calibration for the additional compounds are the same as those listed in the Method 8240 SOP. The concentration is adjusted for either the soil or water analysis. See the continuing calibration standard preparation.

This gives 2 mLs of solution which is stored in a 1.5 - 2.0 mLs amber teflon-lined screw-capped vial at -10°C in the dark. All standard preparation is recorded in the Volatiles Standard Logbook. The Working 5-Point Solution is prepared every two weeks. See above for label information.

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Stock Purgeables Supelprime™ - HC Calibration Standards

Volatile Organic Compounds Mix #1 Catalog No. 4-8775

This mixture contains 2000 ug/mL of each of the following components in methanol:

Chlorobenzene	4-Chlorotoluene
p-Xylene	tert-butylbenzene
o-Xylene	sec-Butylbenzene
Isopropylbenzene	1,3-Dichlorobenzene
n-Propylbenzene	1,4-Dichlorobenzene
2-Chlorotoluene	1,2-Dichlorobenzene

Volatile Organic Compounds Mix #2 Catalog No. 4-8777

This mixture contains 2000 ug/mL of each of the following components in methanol:

Benzene	Toluene
Bromobenzene	1,2,3-Trichlorobenzene
n-Butylbenzene	1,2,4-Trichlorobenzene
Ethylbenzene	1,2,4-Trimethylbenzene
p-Isopropyltoluene	1,3,5-Trimethylbenzene
Naphthalene	m-Xylene
Styrene	

Volatile Organic Compounds Mix #3 Catalog No. 4-8779

This mixture contains 2000 ug/mL of each of the following components in methanol:

1,2-Dibromo-3-chloropropane	Hexachlorobutadiene
1,2-Dibromoethane	1,1,1,2-Tetrachloroethane
1,2-Dichloroethane	1,1,2,2-Tetrachloroethane
1,2-Dichloropropane	1,1,2-Trichloroethane
1,3-Dichloropropane	Trichloroethylene
1,1-Dichloropropylene	1,2,3-Trichloropropane
1,3-Dichloropropylene*	

*Mixture of cis-1,3-Dichloropropylene and trans-1,3-Dichloropropylene, 4000 ug/mL total.

Volatile Organic Compounds Mix #4 Catalog No. 4-8786

This mixture contains 2000 ug/mL of each of the following components in methanol:

Bromochloromethane	1,1-Dichloroethane
Bromoform	2,2-Dichloropropane
Carbon tetrachloride	Tetrachloroethylene
Chloroform	1,1,1-Trichloroethane
Dibromomethane	

Volatile Organic Compounds Mix #5 Catalog No. 4-8797

This mixture contains 2000 ug/mL of each of the following components in methanol:

Bromodichloromethane	cis-1,2-Dichloroethylene
Dibromochloromethane	trans-1,2-Dichloroethylene
1,1-Dichloroethylene	Methylene Chloride

Volatile Organic Compounds Mix #6 Catalog No. 4-8799

This mixture contains 2000 ug/mL of each of the following components in methanol:

Bromomethane	Dichlorodifluoromethane
Chloroethane	Trichlorofluoromethane
Chloromethane	Vinyl Chloride

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The 5-point standards are prepared just prior to calibration. These are used once and are not stored. The following recipe is used in preparation. Each level is a separate solution in a gas-tight syringe.

Waters: 25-mL Purge Volume

Concentration (ppb)	Stock Water 5-pt. Std. (uL)	Working ISS/SSS (uL)	Water Vol. (mLs)
20	20	5	25
15	15	5	25
10	10	5	25
5	5	5	25
1	1	5	25

Soils: 5-mL Purge Volume

Concentration (ppb)	Stock Soil 5-pt. Std. (uL)	Working ISS/SSS (uL)	Water Vol. (mLs)
200	10	5	5
150	7.5	5	5
100	5	5	5
50	2.5	5	5
10	1	5	5

8.5
Stock Matrix Spike Solution

The matrix spike compounds are obtained as neat standards from Supelco. See Neat Standard Logbook for references. The following matrix spike compounds are used:

Benzene	1,1-Dichloroethene
Chlorobenzene	Toluene
Trichloroethene	

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These are stored at approximately -10°C in the dark prior to use. Neat standards are kept for a period of one year or manufacturers date. Upon opening, the contents are transferred to amber vials. The transfer is recorded in the Standard Preparation Logbook. The standard is assigned an SRN. Labeling information is as above.

8.5.1 The STOCK matrix spike solution is prepared as follows:

Compound	Weight	Vol. (MeOH)	Conc.
Benzene	100 mg	10 ml	10,000 ppm
Chlorobenzene	100 mg		
Trichloroethene	100 mg		
1,1-Dichloroethene	100 mg		
Toluene	100 mg		

This is stored in a 3.5 - 5.0 mL amber teflon-lined screw-capped vial at approximately -10°C in the dark. All standard preparation is recorded in the Volatile Standards Logbook. Matrix spike stock solutions are kept for a period of 6 months or until QC indicates a new solution is required. See above for label information.

8.5.2 The WORKING matrix spike solution is prepared as follows:

Compound	Volume	Vol. (MeOH)	Conc.
Stock Matrix Soln.	25 uL	10 mL	25 ppm

This solution is stored at approximately -10°C in several 1.5 -2.0 mL amber teflon-lined screw-capped vials. All standard preparation is recorded in the Volatile Standard Logbook. Working matrix spike solutions are used for a period of 3 months or until low recoveries of the matrix spike compounds indicate a new solution is needed. See above for label information.

Addition of 10 uL of this solution to a sample results in all spike compounds at 50 ppb or 10 ppb (soil/water).

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8.5.3 QC Charting

The QC matrix spike is prepared the same way as the standard by a different analyst and is a separate solution. A BS and BSD is completed once a month.

8.5.4 Stock BFB Solution

The BFB standard is purchased as a neat solution from Supelco.

Compound	Catalog No.	Concentration
Bromofluorobenzene	4-8083M	2000 ppm

The standard is stored at approximately -10°C in the dark and kept for a period of 1 year until opening. Upon opening, the solution is transferred to a 1.5 - 2.0 mL vial and assigned an SRN.

Stock	Amount (uL)	MeOH (uL)	Concentration
BFB	25 uL	1975 uL	25 ppm

Addition of 2 uL/5 mLs results in a concentration of 25 ppb.

All preparation is recorded in the Standards Preparation Logbook. All labels are completed as above. The Working BFB Solutions are kept for a period of 6 months.

NOTE: Intermediate and Working Solutions are never assigned an expiration date exceeding the expiration date of the neat/stock standards/solutions.

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8.6 Reagents**8.6.1 Reagent Water**

Approximately two (2) liters of water is continuously purged with pre-purified nitrogen. The two jars are alternately used. The reagent water is routinely demonstrated to be interference-free. All compounds are less than their EQL or 5X EQL for specific compounds.

8.6.2 Methanol

Purge and Trap B&J Methanol interference-free. Each lot number of methanol is checked for contamination prior to laboratory use and is documented in the QA/QC department.

9.0 PROCEDURE

Before an instrument is used as a measuring device, the instrument response to known reference materials must be determined. The manner in which various instruments are calibrated depends on the particular type of instrument and its intended use. All sample measurements must be made within the calibration range of the instrument. Preparation of all reference materials used for calibration is documented.

9.1 Calibration

An initial 5-point calibration is completed:

- as needed-continuing calibration can not be met
- after a source cleaning and/or column change
- any time a major repair or change has occurred with the instrument that affects calibration

Confirm that the GC/MSD or bench-top is stable and equilibrated. If at all possible, allow the instrument to equilibrate overnight at all operating temperatures if the source/column has been cleaned/changed. Prior to beginning initial calibration it is a good idea to:

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- check the background of air/water levels and base ion by scanning for appropriate ions and also visually inspecting the spectrum scan for any other possible and undesirable background.

- recheck the multiplier settings, after a source is cleaned the EM can most often be dropped.

9.2 PFTBA Autotune or Manual Tune

The instrument is first tuned in one of two ways: autotune or manual tune. The ion abundances in the calibration gas are best monitored near the temperature of analysis of BFB. Monitoring at this temperature produces the most representative cal gas scan and therefore the best estimate of BFB response.

9.2.1 If an AUTOTUNE is to be done, continue below. If not, skip to step 4. An autotune is not run before every 5-point calibration. If the instrument has been down for any reason previously listed or major difficulties in manual tune are encountered, an autotune is performed. Autotunes are generally NOT performed when an existing 5-point is being met.

9.2.2 Enter MSCAL (or appropriate command) to begin the autotune program. Key masses are 69, 219 and 502. A good explanation of the 10 steps and any help needed for any of the screens can be found in the Hewlett-Packard RTE-6/VM Data System Manual.

9.2.3 Follow instructions and retrieve a hardcopy of the autotune results. Check the following:

- passed/fail: in itself, not necessarily an indication of MS performance
- repeller and ion focus settings
- electron multiplier voltage

The repeller and EM voltages are good indicators of the sources' cleanliness. Generally, the lower the setting the cleaner the source. Other factors may however, supersede (i.e. the age of the multiplier) and a clean source will not always autotune these low. The EM is set by autotune program to produce an abundance for mass 69 around 15000. The operator can plan on having to increase this by 100-200 to achieve normal analysis sensitivity.

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Observe peak shape, absence of lead-ons/tailing, the resolution between isotopes, peak width and mass axis. A hardcopy of the profile scan is desirable, and can be filed with the autotune results.

9.2.4 If an AUTOTUNE has just been performed, continue here. If not, skip to step 7. Enter MANUAL TUNE and read the autotune (which was automatically stored in a file). For volatiles, edit the scan parameters to monitor ions 69, 131 and 219.

9.2.5 Enter one of several methods available and adjust the parameters (usually the ion focus, entrance lens and amu gain) to achieve the following relative abundances:

Mass	Relative Abundance
69	100%
131	32-40%
219	35-45%

These will vary with the MS. Mass 219 is usually 5-9% greater than mass 131. Adjust the EM voltage up 100 volts. If necessary, adjust the amu gain for peak shape and high-end isotope resolution. An overall peak-width of 0.500 is desirable.

Again, these adjustments and relative abundances may not guarantee that BFB will meet requirements, but is a good place to start.

9.2.6 Hardcopy the profile scan. This should be filed with the autotune results. This file can serve as a diagnostic tool and can also provide a starting point in the event the operator has trouble meeting the 5-point calibration.

NOTE: In volatiles, as opposed to semi-volatiles, there are a few more things that will affect continuing calibration. Although there may be times when adjusting the tune parameters may help, it will most likely be another problem (i.e. age of standards).

Save the changes to the appropriate Tune File. Exit the program.

9.2.7 If an AUTOTUNE has not been performed, enter MANUAL TUNE and adjust any parameters, if need be. Adjustment may not be necessary, and not desirable, if problems in tuning or meeting the initial calibration have not been encountered. Hardcopy a profile scan and exit.

NOTE: Again, this step in volatiles is not as critical as it is in semi-volatiles. It may be desirable if the operator has been having problems with the instrument, but this is not always necessary. Observing and hardcopying the profile scan does serve as a reference for the next day.

9.3 BFB Analysis

Once the instrument is tuned, a 25 ng injection of 4-Bromofluorobenzene must meet criteria. The BFB can be purged or directly injected. The mass spectrum must meet the following criteria:

Mass	Ion Abundance
50	15-40% of mass 95
75	30-60% of mass 95
95	Base Peak, 100% rel. abund.
96	<2% of mass 95
173	<2% of mass 174
174	>50% of mass 95
175	5 - 9% of mass 174
176	>95% but <101% of mass 174
177	5 - 9% of mass 176

The BFB is analyzed by one of the methods in Appendix A. (These are examples, as methods may be adjusted.) Typical Tekmar conditions also appear in Appendix A. The EM voltage should be 100-200 volts above autotune. The abundances of the designated masses above **MUST** meet the criteria before analyses can begin. If necessary, enter MANUAL TUNE and adjust parameters. The instrument is tuned about every 12 hours of analysis.

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 9.4 Initial Calibration

Each 5-point calibration standard is analyzed according to one of the methods in Appendix A. These are examples. The EM voltage should be 100-200 volts above autotune.

Allow standards to come to ambient temperature.

Fill five 5 mL or 25 mL luer-lock gas-tight syringes with reagent water to overflowing. Replace the plunger and invert. Adjust to 5 mLs (or 25 mLs), confirming the absence of any air bubbles. Pull back slightly on the plunger to allow addition of standards.

Use the following as a guide:

Waters: 25-mL Purge Volume

Concentration (ppb)	Stock Water 5-pt. Std. (uL)	Working ISS/SSS (uL)	Water Vol. (mLs)
20	20	5	25
15	15	5	25
10	10	5	25
5	5	5	25
1	1	5	25

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Soils: 5-mL Purge Volume

Concentration (ppb)	Stock Soil 5-pt. Std. (uL)	Working ISS/SSS (uL)	Water Vol. (mLs)
200	10	5	5
150	7.5	5	5
100	5	5	5
50	2.5	5	5
10	1	5	5

The same tables appear in the standard section. Immediately add the standards to a clean and baked purge vessel. Following the parameters in Appendix A, analyze the 50 ppb or 10 ppb standards. A normal standard will appear very similar to the one in Figure 4. Quantitate the standard against the appropriate ID file. A short list example of one ID file appears in Appendix B. Note the area of the first internal standard. Too low an area will almost guarantee poor/unsatisfactory responses of low-response compounds and too high an area will result in saturation of some compounds at higher levels, resulting in false low response factors at high concentrations.

Response factors are calculated by the data system as follows:

$$RF = \frac{A_x \times Q_s}{A_s \times Q_x}$$

Where:

- A_x = ion abundance for analyte
- A_s = ion abundance for its internal standard
- Q_x = concentration of its internal standard
- Q_s = concentration of analyte

(Response Factors have no units)

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The appropriate quant ion must be in the ID file. See an example of a full-listed ID file in Appendix B. A listing of the target compounds with their appropriate internal standards also appears in Appendix B. Confirm the presence of all targets and the separation of non- co-eluting compounds. Note the response factors for the gasses. If necessary, prepare new standards.

If adjustments to the acquisition parameters are necessary, make them and re-analyze the 50 ppb or 10 ppb standard.

After a good standard is analyzed it is desirable to update the RF's in the ID file (QCAL). This 1) will tell the operator at a glance if one or more standards is low/high and 2) could indicate that one or two were made incorrectly (not necessarily the same thing.)

Analyze all remaining standards. Hardcopy and observe the above. Produce a Form 6 from the data system. Confirm that 1) all CCC's are below 30% and 2) the RF's for all SPCC compounds are >0.300 (Minimum RF for Chloromethane, Bromoform and 1,1,2,2-Tetrachloroethane is 0.100.) An example of an acceptable initial calibration appears in Appendix B. The BFB tune, all standard raw data and Forms 5 & 6 from LIMS are kept in a file. Each instrument has its own initial calibration.

9.5

Daily or Continuing Calibration

Continuing calibration occurs prior to analysis.

NOTE: If time remains after the initial calibration, and the 50 ppb or 10 ppb standard meets continuing calibration criteria, samples can be analyzed up to the 12 hour tune limit. The samples are quantitated against the 50 ppb or 10 ppb standard, unless client specific method requires differently.

After having satisfied BFB tune requirements, a continuing calibration standard must be analyzed. Analyze a 50 ppb or 10 ppb standard following the procedure outlined above. Confirm (CBCHK or Form 7) that all CCC's are <25% and the RF's for all SPCC's are >0.300 (Minimum RF for Chloromethane, Bromoform, and 1,1,2,2-Tetrachloroethane is 0.100.) If so, the continuing calibration is acceptable and analysis can begin.

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NOTE: If additional compounds are required, these are analyzed at the 10 or 50 ppb level only (single point calibration.)

NOTE: In most cases, all CCC's and SPCC's MUST meet requirements. Unless specified either by client-requested method or QC there may be times when analysis will proceed when continuing calibration does not meet criteria. This is approved before analysis.

The response factors are updated in the ID file. The BFB tune and continuing calibration data is kept together in a folder with all sample data analyzed within the next 12 hour period.

If continuing calibration can not be met, either new standards and/or a new 5-point calibration is needed.

9.6 Analysis

9.6.1 Method Blank

Prior to any analysis, the reagent water must be shown to be free of interferences and target compounds.

A 5 mL or 25 mL portion of reagent water is analyzed using one of the methods in Appendix A. All target compounds must be less than the quantitation limit (or <5X the quantitation limit for the following compounds: acetone, dichloro-methane, 2-butanone and toluene.)

Note: On some occasions, acetone and dichloromethane may be allowed at higher levels. IF such is the case, it is approved before analysis and documented in a case narrative.

9.6.2 Once the method blank analysis is complete and acceptable, analysis can proceed. The raw data is kept in the same folder as above.

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9.6.2 Sample Analysis9.6.2.1 Waters

Allow samples and standards to come to ambient temperature.

Remove the plunger from a 5 mL (or 25 mL) luer-lock gas-tight syringe and fill to near over-flowing. Replace the plunger, invert the syringe, and adjust the volume to 5 mLs (or 25 mLs). Confirm the absence of all air bubbles.

Draw back slightly on the plunger. Add 5 uL of the working ISS/SSS solution. Immediately add the sample to a clean purge vessel. Using the methods described in Appendix A analyze the sample. Immediately after analysis, pH check the sample. Record the pH check in the appropriate column in the instrument logbook.

Note: If lower detection limits are required, a 25 mL sample volume will be used.

If a batch is going to be analyzed, which is usually the case, load all samples following the procedure above. After the batch is loaded, replace all samples and standards back in storage.

If dilutions are to be made, the appropriate sample volume is added directly to an appropriate amount of reagent water in the 5 mL (or 25 mL) syringe. If the required amount of sample is less than 1 uL (or < 1 mL), an initial dilution is made first, and a portion of this is added to the reagent water/ISS/SSS mix in the 5 mL (or 25 mL) syringe.

Using those parameters listed in Appendix A, analyze all samples. After analysis, remove the purge vessel from the Tekmar, rinse the purge line and vessel, and place the vessel in the oven to bake at 100°C for at least an hour.

Opened sample vials are used only once unless: 1) any necessary dilutions/reruns are done the same day or 2) there are no other vials for that sample.

9.6.2.2 Soils

Before weighing any samples, check the balance using the appropriate class weights. Record the actual weights in the Balance Logbook. If a discrepancy occurs, contact quality control.

Allow samples and standards to come to ambient temperature.

Weight out 5 grams of the sample into a clean previously baked purge vessel. Record the weight to 0.1 g. Place the vessel on the Tekmar. Add reagent water to overflowing to a 5 mL syringe. Replace the plunger, invert the syringe, and with tapping, adjust to 5 mLs. Confirm there are no air bubbles. Add 5 uL of the working ISS/SSS solution. Transfer the contents to the purge vessel. Using the methods described in Appendix A, analyze the sample. Standards and samples for soil analyses are analyzed in the same manner.

If a batch of samples is to be analyzed, prepare each as above. After the batch is loaded, replace all samples and standards in storage.

If dilutions are to be made, use the following guidelines:

Dilution	Weight	Vol. MeOH Extract
1/2	2.5 g	----
1/5	1.0 g	----
1/10	0.5 g	----
1/20	5.0 g/5 mLs MeOH	250 uL
1/50	5.0 g/5 mLs MeOH	100 uL
1/100	5.0 g/5 mLs MeOH	50 uL
1/200	5.0 g/5 mLs MeOH	25 uL
1/500	5.0 g/5 mLs MeOH	10 uL
1/1000	5.0 g/5 mLs MeOH	5 uL
1/5000	5.0 g/5 mLs MeOH	1 uL

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If a larger dilution is required, either a 1/2 or 1/5 extract is made first, or an intermediate dilution is made from the 1/1 extract first. This intermediate dilution is then taken into account. The Vol. MeOH Extract above is the amount added to an appropriate amount of reagent water/ISS/SSS in the 5 mL syringe, for a 1/1 extract. This is then transferred to the purge vessel on the Tekmar. A volume of methanol is added to each blank associated with diluted soil samples.

Using those parameters in Appendix A, analyze all samples in the batch. After analysis, remove the purge vessels from the Tekmar, dispose of the contents, rinse the purge line and vessel, and place the vessel in the oven to bake at 100°C for at least one hour.

Sample vials/jars are only used once unless: 1) any dilutions/reruns are analyzed the same day or 2) there is only one jar for analysis. A portion of any MeOH extract is immediately removed from the 40 mL vial it was prepared in, and stored in a labelled 1.5 - 2.0 mL amber teflon-lined screw-capped vial kept at approximately -10°C in the dark.

The preparation and analysis is recorded in the GC/MS Volatiles Logbook, and must be completed for each days analysis. An example of a logbook page appears in Appendix C. In addition, a Tune Tracking Form is also filled out for each batch. This form will track the status of the sample once it has been analyzed. This form stays with all the data for that 12 hour period. After final review and submission for the last sample in that 12 hour batch, the quality control is removed from the system and the Tune Form is filed.

In addition, in volatiles, all samples logged into the department appear on both a hold-time summary and a Sample Tracking sheet. The Sample Tracking sheet is used in conjunction with the hold-time summary and the Tune Form to track every single sample in-house. Examples of the Tune Form and the Sample Tracking sheet appear in Appendix C.

Any sample that contains targets above the calibration range is diluted to accurately quantitate those compounds. Any sample that has shown from past experience to be high in background/targets, or appears to be so, is first screened by GC/FID. The intensity of the signal of the non-internal standard peaks is compared to that of the internal standards to give a reasonable estimation of an appropriate dilution.

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9.6.2.3 Drum/Waste Samples

- Non-methanol Miscible

These are treated as soil samples. Extracts are prepared as above.

- Methanol Miscible

A dilution is made directly into methanol. A portion of this is then added to the reagent water/ISS/SSS mixture in the 5 mL syringe. The initial dilution is taken into account when calculating the final dilution factor. A volume of methanol is added to each blank associated with diluted drum samples.

10.0 CALCULATIONS

10.1 Computer Data Production/Reduction

The RTE/Data System produces a Total Ion Chromatogram (TIC), header, quant report and background subtracted spectra. For those clients requiring it, a 10 tentatively identified compound search is also performed. The data system will produce an integration listing and tentative identification of each hit found at the selected percentage of the largest peak present. All data for each sample is kept together. All data for all samples run during that 12 hour period remains together until operator data workup begins. At which time all appropriate QC is photocopied for each RFW# batch in that run batch.

10.1.1 Quantitation of Target Compounds

Quantitation of the target compounds is performed by the data system and LIMS but can be accomplished as follows:

WATERS:

$$\text{concentration (ug/L)} = \frac{[A_x \times I_s]}{[A_{is} \times RF]} \times DF$$

Where:

A_x = area of characteristic ion for target
 I_s = concentration of internal standard (ng)
 A_{is} = area of characteristic ion for int. std.
 RF = response factor for target
 DF = dilution factor (if any)

SOILS:

$$\text{concentration (ug/kg)} = \frac{[A_x \times I_s]}{[A_{is} \times RF \times D]} \times DF$$

Where:

All variables are equal and

D = (100 - % moisture in sample/100) or 1 for wet weight. (As in the case of drum samples)

The sample volume is considered to be "constant" for calculation purposes, and is always entered as five (5) in the Lotus Spreadsheet. Less sample volume (in the case of waters) and extract volume (in the case of soils/drums) are taken into account in the dilution factor at LIMS.

10.1.2

Quantitation of TIC's (Tentatively Identified Compounds)

Quantitation of TIC's is performed by the RTE Data system, operator and LIMS. The formulas above for waters and soils can be used with the following modifications. A_x and A_{is} should be taken from the total ion integration listing accompanying the TIC report produced by the data system. The nearest non-interfered with internal standard should be used. The RF is assumed to be one (1). The concentration is therefore an estimate and is flagged as such with a "J". Any TIC also found in the method blank is flagged with a "JB". With the present LIMS system, the calculations are much simpler.

$$\text{Total ng of TIC} = \frac{\text{**area of TIC} \times \text{ng of its ISTD (50)}}{\text{area of that ISTD}}$$

** The TIC program (*TRQAL or SMLTIC) has been modified to draw the integrated chromatogram in two portions. The operator should visually confirm that the integration is correct. If not, the peak in question must be manually integrated. The total ng of TIC is entered into a spreadsheet at LIMS which then takes into account total solids and the dilution factor in the final calculation of the TIC concentration.

10.2 Operator Data Reduction/Review

The operator reviews the data produced by the RTE system and 1) makes judgements concerning the "realness" of those target compounds found and 2) makes judgements concerning the identification of the tentatively identified compounds and 3) modifies the output to produce a data package reflective of those decisions.

10.2.1 Initial Review

Operator determines that the analysis in itself is acceptable. This means:

QC Samples/Criteria are met

- The method blank contains no interferences or target compounds at the EQL or 5X the EQL for previously listed compounds.
- ALL surrogates are in control in the blank
- ALL surrogates in samples are in control (current surrogate limits are listed below)
- LCS recoveries are advisory only as in-house data is being generated, and will be used to set limits (current limits are listed below)

Water Surrogate Limits: 80 - 120%

Soil Surrogate Limits: Refer to SW-846 Method 8240

Water Matrix Spike Limits: 80 - 120%

Soil Matrix Spike Limits: Refer to SW-846 Method 8240

The sample does not require any further dilutions or analysis at a more concentrated level. Visually confirm complete integration for any large and/or saturated target compounds. Dilutions are made to keep the target in the upper half of the calibration range. The MS and MSD are never diluted to get spiked or non-spiked compounds within range, as this would reduce the matrix affect assessment.

The sample does not require re-analysis for any other reason (i.e. leak, analysis past tune time, ISTD areas low, etc...).

10.2.2 Identification of Targets

The following guidelines are used in the positive identification of target compounds.

"elution of component at the same relative retention time as the standard component."

The elution times should compare within 0.06 min. The standard must be run on the same 12 hour period as the sample. If co-eluting analytes interfere with the comparisons of retention times, other ions characteristic to that compound can be used to confirm relative retention times.

10.2.3 "correspondence of the sample component and standard component mass spectrum." Comparisons of sample spectra to standard spectra must be made using standard spectra obtained from the GC/MS system.

All ions present in the standard spectrum at a 10% relative intensity (most abundant ion being 100%) **MUST** be present in the sample.

The relative intensities of the above ions must agree within $\pm 20\%$, between the standard and sample. If an ion is 50% intensity in the standard the corresponding ion must be between 30 and 70% in the sample.

Ions $> 10\%$ in the sample but not present in the standard must be considered and accounted for. (A user program exists to aid in this).

Operator judgement. If a compound can not be verified by the above, but in the operators technical judgement the ID is correct, it is reported as such.

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10.2.4 Once all positive identification is made, the output file is modified to reflect these decisions (the integrity of the original file is not destroyed). In each case where the file has been edited or manual integration has taken place, the operator must identify, initial and date the changes. An ascii file is created for later transfer to LIMS. All analytes at concentrations less than the Estimated Quantitation Limit are automatically flagged with a J.

10.2.5 Identification of TIC's

In general, up to as many as 10 non-target compounds are tentatively identified by the data system and operator. Compounds with responses < 10% of the nearest ISTD are not identified. The data system provides the operator with a SUB ADC C sample spectrum, spectra of the first three matches and a listing of two other possibilities. Molecular formulas, molecular weights and CAS #'s are included. The following guidelines are used:

10.2.6 Relative intensities of major ions in the reference spectrum should be present in the sample (ions > 10%).

- Relative ions should agree within $\pm 20\%$.
- Molecular ions in the reference should be in the samples.
- Review the possibility of background and/or co-eluting compounds for those ions present in the sample but not in the standard.
- If ions are present in the sample but not in the standard, review the possibility of the presence of background or co-eluting compounds.
- If ions are present in the standard but not in the sample, review the possibility that the ions were subtracted out because they are also common to the background or co-eluting compounds.
- In the event no valid interpretation can be made, the compound is called "unknown".

Interpretation can be often narrowed down to a class of compounds, molecular formula or weight.

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10.3 Final Review

Once 1) the analysis is determined to be acceptable and 2) the initial review and data reduction has occurred (verifiable on the Tune Form) 3) the results transferred to LIMS as ascii files and 4) all forms are produced, the package is put together and submitted for its first 100% review. The checklist in Appendix D is used as an aid to review.

In actuality, some of the forms will not print correctly or at all if many of the review checks are not correct. Some of the checks are performed even as the operator is working up the data to the level accepted and used by LIMS. In this manner, the data is being reviewed as soon as it is produced by the data system.

Upon the first 100% review, the review form is initialed and dated as reviewed. The package, with its review sheet, comments and any C/A forms is submitted to the unit leader or section manager for a second review and validation. Once the data passes review and report generation 1) archival is verified or completed at that time (verification available from the VOA archive logbook) or later and 2) the files are purged from the data system. A hardcopy of the purge file is obtained, initialed and dated and serves as documentation.

The complete analysis scheme can be summarized below (Section 11.1 & 11.2) and in Appendix E. The entire sample tracking system can be summarized in Appendix E.

11.0 QUALITY CONTROL

Quality Control is accomplished through 1) daily tuning and calibration checks and 2) preparation QC traceable through individual extraction batches.

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 11.1 Initial Calibration

PFTBA			
BFB TUNE		Prior to 5-Point Curve	*control chart
200 or 20 ng \			
150 or 15 ng			
100 or 10 ng	5-Point	Need dependent on	*control chart
50 or 5 ng		situation. See	
10 or 1 ng /		Section 9.1	

 11.2 Daily Analysis

PFTBA		
BFB	Prior to continuing calibration	*control chart
Daily Calibration Standard	Prior to samples	*control chart

Samples *

*Any given 12 hour period contains a tune, standard, blank and blank spike. Preparation QC is at a 5% frequency. Instrumental controls are outlined above and further discussed in the procedure section.

<u>Prep QC</u>	<u>Frequency</u>
Blank	Prior to analysis
LCS	1 per analysis batch
MS/MSD's	at least 1 set in 20
Surrogates	every blank, sample and QC Sample
QC Charting	1 set per month

 11.3 The department will review the quality controls as follows:

11.3.1 At least one method blank and at least one Lab Control Standard (LCS) will be included in each laboratory batch. Regardless of the matrix being processed, the LCS and method blanks will be in an aqueous media.

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- 11.3.2 The method blanks will be examined to determine if contamination is being introduced in the laboratory.
- 11.3.3 The LCS will be examined to determine accuracy and precision.
- 11.3.4 Accuracy will be measured by the percent recovery (%R) of the LCS. The recovery must be in range, as determined by statistical analysis, in order to be considered acceptable. Additionally, %R will be plotted on control charts to monitor method accuracy. (Current limits listed in Section 10.2.1.)
- 11.3.5 Precision will be measured by the reproducibility of both blank spikes and will be calculated as Relative Percent Difference (RPD). Results should be <20%. (Control Limits are generated on a quarterly basis).
- 11.3.6 Surrogate Compounds will be added to every sample to measure performance of the analysis. Results must agree within statistical control limits in order to be considered acceptable. (Current limits listed in Section 10.2.1.)

12.0 CORRECTIVE ACTIONS

Listed below are the steps to be taken when an out-of-control situation occurs. The analyst **MUST**:

- demonstrate that all of the problems creating the out-of-control situation were addressed;
- document the problem and the action that was taken to correct the problem;
- document that an in-control situation has been achieved; and
- receive approval (signature) of the unit leader, section manager or QC personnel prior to release of data associated with the problem.

Discussed below are the suggested and required courses of action when an out-of-control situation has occurred.

All surrogate recoveries are calculated.

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If ANY surrogates are outside limits in the Method Blank, it must be re-analyzed. Analyses CAN NOT proceed until an in-control situation is demonstrated. Re-analyze the blank. If surrogates are still out, the instrument may need to be re-tuned (BFB) and/or another calibration standard analyzed. If the problem persists, further maintenance action may be required (i.e. trap replacement, clean separator).

Before pursuing other measures, check to be sure that:

- calculations are correct
- concentrations of the surrogates in the spiking solution are correct
- the correct amount of ISS/SSS solution was added
- ISS/SSS areas are reasonable

If any surrogates in a sample are outside limits, check the above first. Any sample that has a surrogate out must be re-analyzed. The re-analysis can take the form of a dilution. If the surrogate (s) is/are still outside limits a matrix effect is demonstrated and both reports are submitted.

If all surrogates are in-control on the re-analysis, only the second analysis is reported.

Every effort is made to complete the re-analysis within hold-time. If this is impossible (i.e. capacity hold-times preclude re-analyses hold-time) both reports may be submitted. This is documented in the narrative.

If the sample with the out-of-control surrogates is the same sample on which the MS and MSD has been performed, and the pattern is duplicated, then re-analysis is NOT required. Documentation of the similarities is required.

MS and MSD recoveries and RPD's are advisory only. However, if problems are frequent, investigation of the system is necessary.

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12.1 BFB Criteria

If BFB criteria can not be met, determine if the source of the problem is instrumental or tune related. Inspect overall sensitivity, possible excessive background, the proportionality of the masses, relative abundances of the target masses. If it seems tune related, adjust the tune parameters in Manual Tune slightly, until acceptance is achieved. If the problem seems instrumental, perform suggested trouble-shooting to locate and correct the problem (Suggestions can be found in most of the manuals). NO analysis can proceed until criteria are met. Each instrument will have its own idiosyncracies.

12.2 Initial Calibration

If initial calibration can not be met, determine if the problem is analytical or instrumental. Some suggested questions to ask would be:

- were the standards prepared correctly?
- was the proper amount analyzed?
- check the chromatogram - did something happen on one or two analyses; i.e. a leak
- check the response factors - is one concentration level very high or low? re-analyze it
- how old are the standards?

The course of action depends on the problem

12.3 Continuing Calibration

If continuing calibration can not be met, determine if the problem is analytical or instrumental. Some suggestions:

- check the chromatography
 - is overall sensitivity low?
 - excessive background?
 - how old is the standard?
 - need a new 5-point?
- has the tune wandered?

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Compare the relative abundances of 69, 131 and 219 from that days manual tune to those on the day the initial calibration was analyzed. Slight adjustments to the tune may bring the standard in.

Certain compounds will help indicate what the problem is. The course of action depends on the problem.

12.4 Blank

If the method blank is/appears to be contaminated:

- re-analyze it on a different position

If contamination is still present, the problem may be in one of the common elements, such as the trap, transfer line, port valve or column or the internal standard solution. Baking the trap/column and running position blanks may be necessary. If contamination has occurred beyond that, and maintenance is required (i.e. replace trap) it is documented in the Maintenance Logbook. Analysis CAN NOT proceed until the blank is free of contamination.

12.5 LCS's

Limits must be generated by in-house data. Advisory only. Outliers are documented.

12.6 Matrix Spikes

MS and MSD recoveries and RPD's are advisory only and function to illustrate matrix effects on the efficiency and completeness of target analyses. Outliers are documented.

Maintenance logbooks do exist. Major maintenance and problems are noted in these logbooks. The entry number is transferred to the appropriate run log, initialed and dated. Other courses of action taken for out-of-control situations are documented either on the review form or the case narrative or in the operator logbook itself.

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13.0

HEALTH AND SAFETY

As always, general laboratory safety practices should always be followed. Waste samples should be handled with care due to the uncertainty of the properties and contents involved.

Refer to the specific MSDS for the hazardous properties of ANY chemical or reagent involved in this procedure.

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Table 1
Characteristic Mass (m/z) for Purgeable Organic Compounds

Analyte	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Benzene	78	77, 158
Bromobenzene	156	48, 130
Bromochloromethane	128	85, 127
Bromodichloromethane	83	175, 254
Bromoform	173	96
Bromomethane	94	92, 134
n-Butylbenzene	91	134
sec-Butylbenzene	105	91, 134
tert-Butylbenzene	119	119
Carbon tetrachloride	117	77, 114
Chlorobenzene	112	66
Chloroethane	64	85
Chloroform	83	52
Chloromethane	50	126
2-Chlorotoluene*	91	126
4-Chlorotoluene*	91	155, 157
1,2-Dibromo-3-chloropropane	75	127
Dibromochloromethane	129	109, 188
1,2-Dibromoethane	107	95, 174
Dibromomethane	93	111, 148
1,2-Dichlorobenzene	146	111, 148
1,3-Dichlorobenzene	146	111, 148
1,4-Dichlorobenzene	146	87
Dichlorodifluoromethane	85	65, 83
1,1-Dichloroethane	63	98
1,2-Dichloroethane	62	61, 63
1,1-Dichloroethene	96	61, 98
cis-1,2-Dichloroethene	96	61, 98
trans-1,2-Dichloroethene	96	112
1,2-Dichloropropane	63	78
1,3-Dichloropropane	76	97
2,2-Dichloropropane	77	110, 77
1,1-Dichloropropene	75	106
Ethylbenzene*	91	223, 227
Hexachlorobutadiene	225	120
Isopropylbenzene	105	134, 91
p-Isopropylbenzene	119	86, 49

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Table 1
 (continued)
Characteristic Mass (m/z) for Purgeable Organic Compounds

Analyte	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Methylene chloride	84	86, 49
Naphthalene	128	---
n-Propylbenzene*	91	120
Styrene	104	78
1,1,1,2-Tetrachloroethane	131	133, 119
1,1,2,2-Tetrachloroethane	83	131, 85
Tetrachloroethene	166	168, 129
Toluene*	92	91
1,2,3-Trichlorobenzene	180	182, 145
1,2,4-Trichlorobenzene	180	182, 145
1,1,1-Trichloroethane*	97	99, 61
1,1,2-Trichloroethane*	83	97, 85
Trichloroethene	95	130, 132
Trichlorofluoromethane	101	103
1,2,3-Trichloropropane*	75	77
1,2,4-Trimethylbenzene	105	120
1,3,5-Trimethylbenzene	105	120
Vinyl chloride	62	64
o-Xylene*	106	91
m-Xylene*	106	91
p-Xylene*	106	91
<u>Internal Std./Surrogates</u>		
4-Bromofluorobenzene	95	174, 176
1,2-Dichlorobenzene-d ₄	152	150
Pentafluorobenzene	168	
Chlorobenzene-d ₅	117	
1,4-Difluorobenzene	114	
1,2-Dichloroethane-d ₄	65	
Toluene-d ₈	98	

*Note: The primary and secondary ions listed here are taken directly from SW-846 Method 8260. There are instances where we have chosen different ions (i.e. interferences from co-eluting compounds or for enhances identification purposes.)

Table 2

Estimated Quantitation Limits for Volatile Analytes^a

	Estimated Quantitation Limits		
	Ground Water (ug/L)		Low Soil/Sediment ^b (ug/kg)
Volume of water purged	5 mL	25 mL	
All analytes in Table 1	5	1	5

^aEstimated Quantitation Limit (EQL) - The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The EQL is generally 5 to 10 times the MDL. However, it may be nominally chosen within these guidelines to simplify data reporting. For many analytes the EQL analyte concentration is selected for the lowest non-zero standard in the calibration curve. Sample EQLs are highly matrix-dependent. The EQLs listed herein are provided for guidance and may not always be achievable. See the following information for further guidance on matrix-dependent EQLs.

^bEQLs listed for soil/sediment are based on wet weight. Normally data is reported on a dry weight basis; therefore, EQLs will be higher, based on the percent dry weight in each sample.

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Figure 1**Example of Maintenance Log**WESTON-Gulf Coast, Inc.
GC/MS Volatile Maintenance Log
Instrument 3

Date of Maintenance: _____	Entry No.: _____
Analyst: _____	
Description: _____	

Follow Up: _____	

Analyst: _____ Date: _____	

Date of Maintenance: _____	Entry No.: _____
Analyst: _____	
Description: _____	

Follow Up: _____	

Analyst: _____ Date: _____	

Date of Maintenance: _____	Entry No.: _____
Analyst: _____	
Description: _____	

Follow Up: _____	

Analyst: _____ Date: _____	

Eff. Date: 04/26/93 Initiated By: QC Department Approved By: J.A. Kaczinski Authorized By: A. M. Henry SP No. 21-16G-8260

Figure 2

NEAT GC/MS STANDARDS LOGBOOK					
<u>Neat Standards Ref.#</u>	<u>Lot#</u>	<u>Compound/Kit</u>	<u>Rec'd</u>	<u>Opened</u>	<u>Comments</u>
<p>FIGURE 2. Neat Standards Logbook: This logbook is used to record/track ALL neat standards, either as single compounds or kits, received and used by the GC/MS Department. Neat standard reference numbers are coded by a single letter designating the compound type followed by the date received.</p> <p>S = Surrogate T = Target I = Internal Standard M = Matrix Spike</p> <p>All neat standards are recorded and marked with a code upon receipt, then stored at -10°C until use.</p>					

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Figure 3

GC/MS Standard Logbook

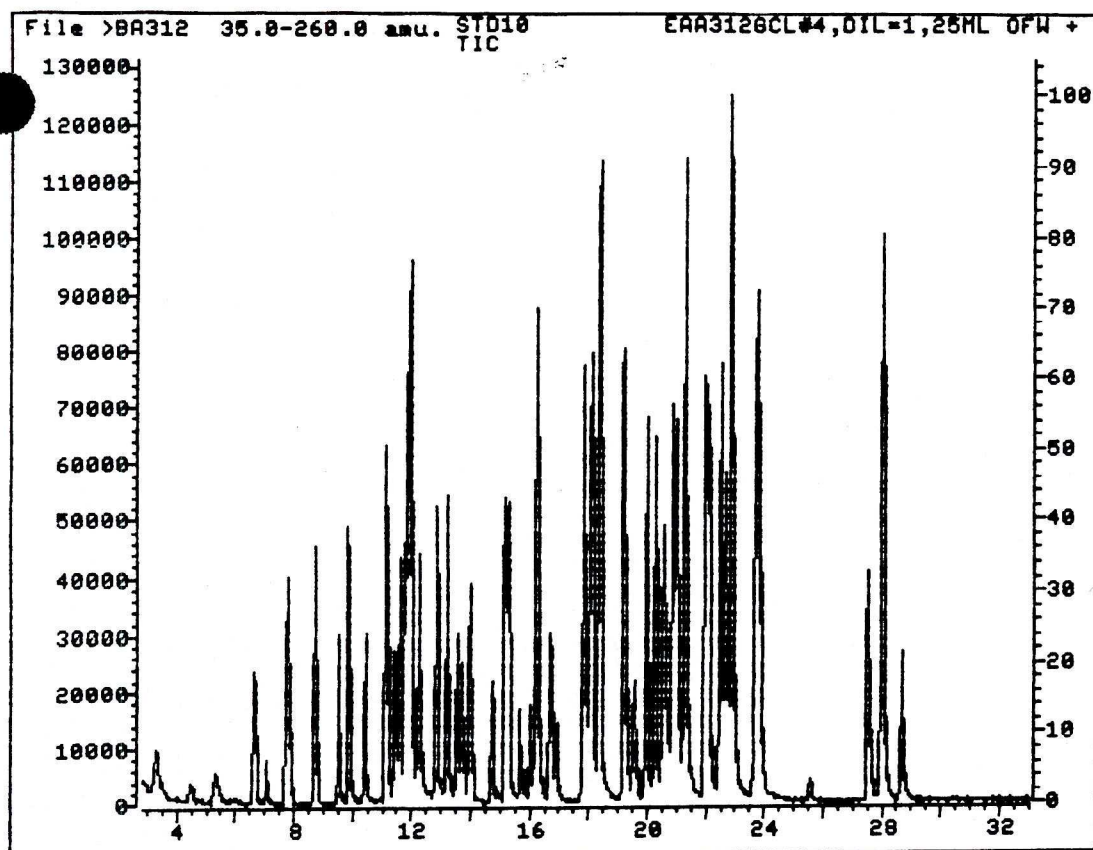
Standard Reference Number (SRN)	Date: _____ Initials: _____ Standard Name: _____	Neat Standard Ref. No.(s): _____
<div style="border: 1px solid black; width: 80px; height: 20px; margin: 10px 0;"></div> <p>Preparation (Lot #'s, weights, solvents)</p>		
<p>Comments: _____</p> <p>_____</p>		
<p>Final Concentration: _____</p>		
<p>Figure 3. VOA Standards Logbook. This logbook is used to recount ALL standard preparations. ALL prepared standards contain a label bearing the following information:</p> <div style="text-align: center; margin-top: 20px;"> <p>Standard Name Concentration Date Prepared Preparers Initials Std. Ref. No. (SRN)</p> </div>		

Eff. Date: 04/26/93 Initiated By: QC Department Approved By: J.A. Kaczinski Authorized By: A. M. Henry SP No. 21-16G-8260

Figure 4

Total Ion Chromatogram for Normal 25 mL Purge Water

Figure 4



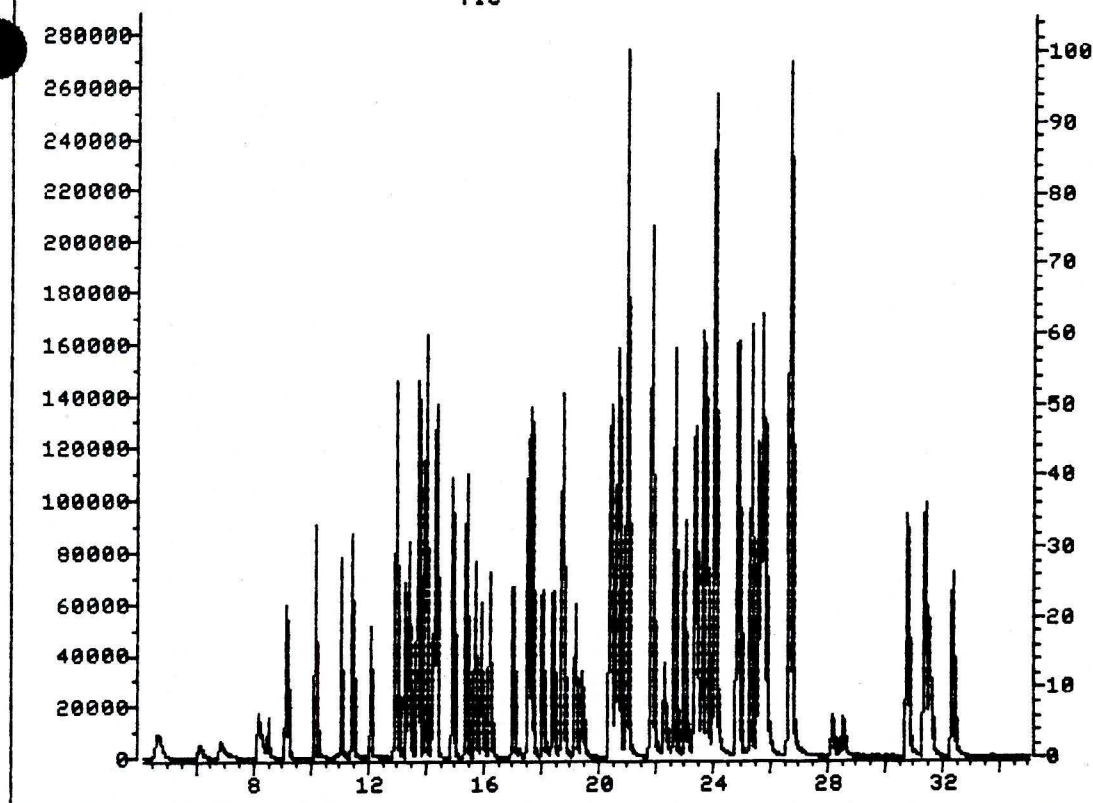
TOTAL ION CHROMATOGRAM FOR NORMAL 35ml PURGE H₂O

MS data file header from : >BA312

Sample: STD10 EAA312 Operator: MANAGER MS 3/12/93 14:51
Misc : GCL#4,DIL=1,25ML OFW + 5UL ISS 545 + 10UL 543
Sys. #: 4 MS model: 70 SW/HW rev.: IA ALS #: 0
Method file: WESTON Tuning file: TUNE4 No. of extra records: 2
Source temp.: 0 Analyzer temp.: 250 Transfer line temp.: 0

Chromatographic temperatures :	-20.	50.	170.	0.	0.
Chromatographic times, min. :	4.0	0.0	17.0	0.0	0.0
Chromatographic rate, deg/min:	15.0	6.0	0.0	.5	0.0

File >LA315 35.0-260.0 amu. STD50 CKA3156CL#3,DIL=1,5ML OFW + 5
TIC



TOTAL ION CHROMATOGRAM FOR NORMAL 56 SCALS

MS data file header from : >LA315

Sample: STD50 CKA315 Operator: MANAGER MS 3/15/93 11:21
Misc : GCL#3,DIL=1,5ML OFW + 5UL ISS 545 + 2.5UL 559
Sys. #: 3 MS model: 96 SW/HW rev.: IA ALS #: 0
Method file: JOHN Tuning file: TUNE3 No. of extra records: 2
Source temp.: 150 Analyzer temp.: 180 Transfer line temp.: 250

Chromatographic temperatures :	-20.	50.	170.	0.	0.
Chromatographic times, min. :	4.0	0.0	17.0	0.0	0.0
Chromatographic rate, deg/min:	15.0	6.0	0.0	0.0	0.0



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APPENDIX A.

Method Listings

Tekmar Conditions

Flow Settings



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METHOD FILE LIST

Method File: DON GC TYPE: 5996 Run Type: SCAN, GC, EI
Column: Cap Splitless: Yes

Temperature: Inj. P Xfer L Source Analyzer FID
160.0 250.0 150.0 180.0 .0

GC/DIP	LEVEL 1	LEVEL 2	LEVEL 3	LEVEL 4	POST RUN
Temp 1	20.0	160.0	0.0	0.0	0.0
Time 1	2.0	5.0	0.0	0.0	0.0
Rate	10.0	0.0	0.0	0.0	
Temp 2	160.0	0.0	0.0	0.0	
Time 2	5.0	0.0	0.0	0.0	

Run Time: 21.00
Scan Start Time: 6.00
Splitless valve time: 0.00

	ON	OFF	ON	OFF
Solvent Diverter Valve:	327.0	327.0	327.0	327.0
Relay #1:	0.10	0.30	327.0	327.0
Relay #2:	0.10	0.30	327.0	327.0
Triac #0:	327.0	327.0	327.0	327.0
Triac #1:	327.0	327.0	327.0	327.0

ALS Operating Conditions

Number of sample washes: 2	Number of Sample Pumps: 6
Solvent A Washes: 2	Solvent B Washes: 0
Sample Viscosity Wait: 0	Injection Mode is FAST

Scan Parameters: Mass Range: 35 - 260
Multiplier Voltage: 1984
Number of A/D Samples: 8
GC Peak Threshold: 10000 counts
Threshold: 15 counts



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METHOD FILE LIST

Method File: ROYF GC TYPE: 5890 Run Type: SCAN, GC, EI
Column: Cap Splitless: Yes

Temperature: Inj. P Xfer L Source
 160.0 250.0 0.0

GC/DIP		LEVEL A	LEVEL B	POST RUN
Temp 1	100.0	200.0	0.0	0.0
Time 1	2.0	5.0	0.0	0.0
Rate	5.0	0.0	0.0	
Temp 2	200.0	0.0	0.0	
Time 2	5.0	0.0	0.0	

Oven Equilibration Time: 0.00 min

Run Time: 15.00
Scan Start Time: 7.00
Splitless valve time: 0.00

	ON	OFF	ON	OFF
Relay #1:	0.10	0.30	327.0	327.0
Relay #2:	0.10	0.30	327.0	327.0
Triac #0:	327.0	327.0	327.0	327.0
Triac #1:	327.0	327.0	327.0	327.0

Scan Parameters: Mass Range: 35 - 260
 Multiplier Voltage: 1606
 Number of A/D Samples: 16
 GC Peak Threshold: 10000 counts
 Threshold: 10 counts



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METHOD FILE LIST

Method File: JOHN GC TYPE: 5996 Run Type: SCAN, GC, EI
Column: Cap Splitless: Yes

Temperature: Inj. P Xfer L Source Analyzer FID
 160.0 250.0 150.0 180.0 .0

GC/DIP	LEVEL 1	LEVEL 2	LEVEL 3	LEVEL 4	POST RUN
Temp 1	0.0	80.0	170.0	0.0	170.0
Time 1	4.0	2.0	10.0	0.0	3.0
Rate	10.0	4.0	0.0	0.0	
Temp 2	80.0	170.0	0.0	0.0	
Time 2	2.0	10.0	0.0	0.0	

Run Time: 28.50
Scan Start Time: 1.50
Splitless valve time: 0.00

	ON	OFF	ON	OFF
Solvent Diverter Valve:	327.0	327.0	327.0	327.0
Relay #1:	0.10	0.30	327.0	327.0
Relay #2:	0.10	0.30	327.0	327.0
Triac #0:	327.0	327.0	327.0	327.0
Triac #1:	327.0	327.0	327.0	327.0

ALS Operating Conditions

Number of sample washes: 2	Number of Sample Pumps: 6
Solvent A Washes: 2	Solvent B Washes: 0
Sample Viscosity Wait: 0	Injection Mode is FAST

Scan Parameters: Mass Range: 35 - 260
 Multiplier Voltage: 1889
 Number of A/D Samples: 8
 GC Peak Threshold: 10000 counts
 Threshold: 20 counts



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METHOD FILE LIST

Method File: WESTON GC TYPE: 5890 Run Type: SCAN, GC, EI
Column: Cap Splitless: Yes

Temperature: Inj. P Xfer L Source
 160.0 250.0 0.0

GC/DIP		LEVEL A	LEVEL B	POST RUN
Temp 1	-20.0	80.0	170.0	0.0
Time 1	4.0	2.0	10.0	0.0
Rate	10.0	4.0	0.0	
Temp 2	80.0	170.0	0.0	
Time 2	2.0	10.0	0.0	

Oven Equilibration Time: 0.50 min

Run Time: 37.00
Scan Start Time: 2.50
Splitless valve time: 0.00

	ON	OFF	ON	OFF
Relay #1:	0.10	0.30	327.0	327.0
Relay #2:	0.10	0.30	327.0	327.0
Triac #0:	327.0	327.0	327.0	327.0
Triac #1:	327.0	327.0	327.0	327.0

Scan Parameters: Mass Range: 35 - 260
 Multiplier Voltage: 1748
 Number of A/D Samples: 16
 GC Peak Threshold: 10000 counts
 Threshold: 10 counts

Eff. Date: 04/26/93 Initiated By: QC Department Approved By: J.A. Kaczinski Authorized By: A. M. Henry SP No. 21-16G-8260

Tekmar Conditions

Trap Temp. Prior to Purge	< 30
Desorb Preheat	100
Desorb	180
Bake	225
Purge Time	10 - 11 min
Desorb	4 min
Bake Time	10 - 11 min

The bake time on instrument #6 is slightly longer

Flow Conditions

Purge Pressure	20 psi
Purge Flow Rate	40 mLs/min

Flow Adjustment

Capillary Column: 5970/5971/MSD's;
5995 Benchtop

- Make-up gas off/separator pump on: flow through separator is 5-10 mLs/minutes.

- Open make-up gas: adjust until you achieve ~30 mLs/minute through the separator. (On MSD's - adjust to 0.5 torr on gauge)

(Flow into the Mass Spec is ≤ 1 mL/minute)

Approximate Vacuums

5971	$\sim 5 \times 10^{-6}$ torr
5970	$\sim 2-3 \times 10^{-5}$ torr
5995	$\sim 1-2 \times 10^{-5}$ torr



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APPENDIX B.

ID File Listings

Initial Calibration Example

Quant ID File: ID_826::U2

Title: ID FILE FOR CAPILLARY METHOD 8260 INST. #4 WATERS

Last EDIT Date: 930313 10:58

Last Calib Date: 930323 11:46

RT Window (+/- min): 1.00
Max Hits/Compound: 5
Minimum Area: 500
Peak/Base Peak Ratio: 40.00 %
Slope Sensitivity: .200
Subtraction Method: 3
Ignore max check: NO
Auto Qdel Method: 5
Units of Conc: ppb

Number of Compounds: 82

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
1)	*Pentafluorobenzene	(I)	11.78(1.000)	1.00000	10.00
2)	Dichlorodifluoromethane	(T)	3.17(.269)	3.31883	10.00
3)	Chloromethane	(T)	4.38(.372)	.90686	10.00
4)	Vinyl Chloride	(T)	5.30(.450)	1.39560	10.00
5)	Bromomethane	(T)	6.57(.558)	2.23762	10.00
6)	Chloroethane	(T)	6.97(.591)	.80301	10.00
7)	Trichlorofluoromethane	(T)	7.69(.653)	5.42562	10.00
8)	1,1-Dichloroethene	(T)	8.63(.732)	1.88998	10.00
9)	Methylene Chloride	(T)	9.41(.799)	1.37767	10.00
10)	Acetone	(T)	10.47(.889)	.48637	10.00
11)	Acrolein	(T)	8.59(.729)	.00014	1600.00
12)	Acrylonitrile	(T)	9.79(.831)	.04288	320.00
13)	trans-1,2-Dichloroethene	(T)	9.76(.829)	1.93441	10.00
14)	1,1-Dichloroethane	(T)	10.31(.875)	3.07527	10.00
15)	2,2-Dichloropropane	(T)	11.04(.937)	2.28096	10.00
16)	cis-1,2-Dichloroethene	(T)	11.06(.938)	1.74699	10.00
17)	Chloroform	(T)	11.49(.975)	3.99303	10.00
18)	Bromochloromethane	(T)	11.35(.963)	.89470	10.00
19)	1,1,1-Trichloroethane	(T)	11.68(.992)	3.71881	10.00
20)	2-Butanone	(T)	11.12(.944)	.13639	10.00
21)	Carbon Disulfide	(T)	8.80(.747)	3.82985	10.00
22)	Iodomethane	(T)	8.78(.745)	8.84562	20.00
23)	Vinyl Acetate	(T)	10.47(.889)	.48229	10.00
24)	*1,4-Difluorobenzene	(I)	12.74(1.000)	1.00000	10.00
25)	Carbon Tetrachloride	(T)	11.88(.932)	.89246	10.00
26)	1,2-Dichloroethane-d4	(S)	12.11(.951)	.22629	10.00
27)	1,1-Dichloropropene	(T)	11.90(.934)	.51409	10.00
28)	Methyl-tert-Butyl Ether	(T)	9.83(.772)	0.00000	10.00
29)	Benzene	(T)	12.17(.955)	.73344	10.00
30)	1,2-Dichloroethane	(T)	12.21(.958)	.23227	10.00
31)	Toluene-d8	(S)	15.05(1.181)	.96588	10.00
32)	Trichloroethene	(T)	13.09(1.028)	.48837	10.00
33)	2-Chloroethyl vinyl ether	(T)	14.41(1.131)	.06056	10.00
34)	1,2-Dichloropropane	(T)	13.44(1.055)	.28915	10.00
35)	Bromodichloromethane	(T)	13.90(1.091)	.71356	10.00
36)	Dibromomethane	(T)	13.62(1.069)	.25532	10.00
37)	4-Methyl-2-Pentanone	(T)	14.93(1.172)	.07122	10.00
38)	trans-1,3-Dichloropropene	(T)	15.62(1.226)	1.27202	10.00
39)	Toluene	(T)	15.17(1.191)	1.01335	10.00

40)	1,2-Dibromoethane	(T)	16.79(1.318)	.29742	10.00
41)	cis-1,3-Dichloropropene	(T)	14.62(1.148)	.34242	10.00
42)	1,1,2-Trichloroethane	(T)	15.91(1.249)	.16938	10.00
43)	*Chlorobenzene-d5	(I)	17.68(1.000)	1.00000	10.00
44)	Tetrahydrofuran	(T)	15.03(.850)	0.00000	10.00
45)	Bis(Chloromethyl)Ether	(T)	13.89(.786)	0.00000	6400.00
46)	2-Hexanone	(T)	16.43(.929)	.07863	10.00
47)	Ethyl methacrylate	(T)	15.80(.894)	.19894	20.00
48)	Tetrachloroethene	(T)	16.10(.910)	1.05323	10.00
49)	1,3-Dichloropropane	(T)	16.18(.915)	.31498	10.00
50)	Dibromochloromethane	(T)	16.59(.938)	.64263	10.00
51)	Chlorobenzene	(T)	17.74(1.003)	1.00923	10.00
52)	1,1,1,2-Tetrachloroethane	(T)	17.92(1.013)	.61154	10.00
53)	Ethylbenzene	(T)	18.00(1.018)	.49399	10.00
54)	p-Xylene	(T)	18.23(1.031)	2.93726	10.00
55)	m-Xylene	(T)	18.23(1.031)	2.93726	10.00
56)	o-Xylene	(T)	19.03(1.076)	1.45111	10.00
57)	Styrene	(T)	19.07(1.079)	.82965	10.00
58)	Bromoform	(T)	19.41(1.097)	.37937	10.00
59)	*1,2-Dichlorobenzene-d4	(I)	23.55(1.000)	1.00000	10.00
60)	Bromofluorobenzene	(S)	20.14(.855)	.97337	10.00
61)	Isopropylbenzene	(T)	19.85(.843)	2.30424	10.00
62)	1,1,2,2-Tetrachloroethane	(T)	20.55(.873)	.31823	10.00
63)	Bromobenzene	(T)	20.42(.867)	.70202	10.00
64)	1,2,3-Trichloropropane	(T)	20.59(.874)	.08397	10.00
65)	n-Propylbenzene	(T)	20.75(.881)	.54787	10.00
66)	2-Chlorotoluene	(T)	20.87(.886)	.53287	10.00
67)	trans-1,4-dichloro-2-butene	(T)	20.60(.875)	.31496	20.00
68)	4-Chlorotoluene	(T)	21.12(.897)	.56787	10.00
69)	1,3,5-Trimethylbenzene	(T)	21.16(.899)	1.91364	10.00
70)	tert-Butylbenzene	(T)	21.86(.929)	1.97576	10.00
71)	1,2,4-Trimethylbenzene	(T)	21.98(.934)	1.80746	10.00
72)	sec-Butylbenzene	(T)	22.37(.950)	2.77682	10.00
73)	p-Isopropyltoluene	(T)	22.73(.965)	2.29034	10.00
74)	1,3-Dichlorobenzene	(T)	22.53(.957)	1.13387	10.00
75)	1,4-Dichlorobenzene	(T)	22.75(.966)	1.09543	10.00
76)	1,2-Dichlorobenzene	(T)	23.59(1.002)	.99663	10.00
77)	n-Butylbenzene	(T)	23.67(1.005)	2.30917	10.00
78)	1,2-Dibromo-3-Chloropropane	(T)	25.45(1.081)	.04389	10.00
79)	1,2,4-Trichlorobenzene	(T)	27.38(1.163)	.76701	10.00
80)	Hexachlorobutadiene	(T)	27.85(1.183)	1.44748	10.00
81)	Naphthalene	(T)	27.93(1.186)	.40273	10.00
82)	1,2,3-Trichlorobenzene	(T)	28.52(1.211)	.53017	10.00

* Compound is ISTD

Quant ID File: ID_826::U2

Title: ID FILE FOR CAPILLARY METHOD 8260 INST. #4 WATERS

Last EDIT Date: 930313 10:58

Last Calib Date: 930323 11:46

RT Window (+/- min): 1.00
Max Hits/Compound: 5
Minimum Area: 500
Peak/Base Peak Ratio: 40.00 %
Slope Sensitivity: .200
Subtraction Method: 3
Ignore max check: NO
Auto Qdel Method: 5
Units of Conc: ppb

Number of Compounds: 82

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
1)	*Pentafluorobenzene	(I)	11.78(1.000)	1.00000	10.00
	Mass Abun				
	168.0 100.00				
	99.0 65.00				
	137.0 13.00				

Quant Ion: ~~137.0~~ 168.0 Ref Namr: <None>
max 4126193

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Label Methods 1-5: N,R,S,#,U
User Label: Pentafluorobenzene

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
2)	Dichlorodifluoromethane	(T)	3.17(.269)	3.31883	10.00
	Mass Abun				
	85.0 100.00				
	87.0 33.00				

Quant Ion: 85.0 Ref Namr: DICLFL

RT Window: <None> Peaks/min: <None>
Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Label Methods 1-5: N,R,S,#,U

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
3)	Chloromethane	(T)	4.38(.372)	.90686	10.00
	Mass Abun				
	49.8 100.00				
	51.8 35.00				

Quant Ion: 49.8 Ref Namr: CLMTHN

RT Window: <None> Peaks/min: <None>
 Minimum Area: <None> Peak/Base error: <None>
 Slope sens: <None> Subtraction Method:
 Ignore max: Auto Qdel Method:

Label Methods 1-5: N,R,S,#,U

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
4)	Vinyl Chloride	(T)	5.30(.450)	1.39560	10.00
	Mass Abun				
	61.8 100.00				
	63.8 32.00				

Quant Ion: 61.8 Ref Namr: UNYCL

RT Window: <None> Peaks/min: <None>
 Minimum Area: <None> Peak/Base error: <None>
 Slope sens: <None> Subtraction Method:
 Ignore max: Auto Qdel Method:

Label Methods 1-5: N,R,S,#,U

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
5)	Bromomethane	(T)	6.57(.558)	2.23762	10.00
	Mass Abun				
	93.8 100.00				
	95.8 92.00				

Quant Ion: 93.8 Ref Namr: BRMTHN

RT Window: <None> Peaks/min: <None>
 Minimum Area: <None> Peak/Base error: <None>
 Slope sens: <None> Subtraction Method:
 Ignore max: Auto Qdel Method:

Label Methods 1-5: N,R,S,#,U

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
6)	Chloroethane	(T)	6.97(.591)	.80301	10.00
	Mass Abun				
	63.8 100.00				
	65.8 32.00				

Quant Ion: 63.8 Ref Namr: CLETHN

RT Window: <None> Peaks/min: <None>
 Minimum Area: <None> Peak/Base error: <None>
 Slope sens: <None> Subtraction Method:
 Ignore max: Auto Qdel Method:

Label Methods 1-5: N,R,S,#,U

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
7)	Trichlorofluoromethane	(T)	7.69(.653)	5.42562	10.00
	Mass Abun				
	101.0 100.00				
	103.0 66.00				

Quant Ion: 101.0 Ref Namr: TRICFM

RT Window: <None> Peaks/min: <None>

Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Label Methods 1-5: N,R,S,#,U

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
8)	1,1-Dichloroethene	(T)	8.63(.732)	1.88998	10.00
	Mass Abun				

	61.0 100.00	Quant Ion:	96.0	Ref Namr: DCE11=	
	96.0 54.00				
	98.0 35.00				
	RT Window: <None>	Peaks/min: <None>			
	Minimum Area: <None>	Peak/Base error: <None>			
	Slope sens: <None>	Subtraction Method:			
	Ignore max:	Auto Qdel Method:			

Label Methods 1-5: N,R,S,#,U

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
9)	Methylene Chloride	(T)	9.41(.799)	1.37767	10.00
	Mass Abun				

	49.0 100.00	Quant Ion:	84.0	Ref Namr: METHCL	
	84.0 70.00				
	86.0 50.00				
	RT Window: <None>	Peaks/min: <None>			
	Minimum Area: <None>	Peak/Base error: <None>			
	Slope sens: <None>	Subtraction Method:			
	Ignore max:	Auto Qdel Method:			

Label Methods 1-5: N,R,S,#,U

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
10)	Acetone	(T)	10.47(.889)	.48637	10.00
	Mass Abun				

	42.8 100.00	Quant Ion:	42.8	Ref Namr: ACETON	
	57.8 27.00				
	RT Window: <None>	Peaks/min: <None>			
	Minimum Area: <None>	Peak/Base error: <None>			
	Slope sens: <None>	Subtraction Method:			
	Ignore max:	Auto Qdel Method:			

Label Methods 1-5: N,R,S,#,U

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
11)	Acrolein	(T)	8.59(.729)	.00014	1600.00
	Mass Abun				

	56.0 100.00	Quant Ion:	56.0	Ref Namr: ACROLE	
	55.0 Display only				
	65.0 Display only				

Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Label Methods 1-5: N,R,S,#,U

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
12)	Acrylonitrile	(T)	9.79(.831)	.04288	320.00
	Mass Abun				
	53.0 100.00				
	52.0 Display only				
	51.0 Display only				
	Quant Ion: 53.0		Ref Namr: ACRYLO		
	RT Window: <None>		Peaks/min: <None>		
	Minimum Area: <None>		Peak/Base error: <None>		
	Slope sens: <None>		Subtraction Method:		
	Ignore max:		Auto Qdel Method:		

Label Methods 1-5: N,R,S,#,U

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
13)	trans-1,2-Dichloroethene	(T)	9.76(.829)	1.93441	10.00
	Mass Abun				
	61.0 100.00				
	96.0 61.00				
	98.0 40.00				
	Quant Ion: 96.0		Ref Namr: TDCE12		
	RT Window: <None>		Peaks/min: <None>		
	Minimum Area: <None>		Peak/Base error: <None>		
	Slope sens: <None>		Subtraction Method:		
	Ignore max:		Auto Qdel Method:		

Label Methods 1-5: N,R,S,#,U

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
14)	1,1-Dichloroethane	(T)	10.31(.875)	3.07527	10.00
	Mass Abun				
	63.0 100.00				
	65.0 31.00				
	Quant Ion: 63.0		Ref Namr: DCE11		
	RT Window: <None>		Peaks/min: <None>		
	Minimum Area: <None>		Peak/Base error: <None>		
	Slope sens: <None>		Subtraction Method:		
	Ignore max:		Auto Qdel Method:		

Label Methods 1-5: N,R,S,#,U

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
15)	2,2-Dichloropropane	(T)	11.04(.937)	2.28096	10.00
	Mass Abun				
	77.0 100.00				
	97.0 20.00				
	Quant Ion: 77.0		Ref Namr: VOA12		

Minimum Area: <None> Peak/Base error: <None>
Slope sens: <None> Subtraction Method:
Ignore max: Auto Qdel Method:

Label Methods 1-5: N,R,S,#,U

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
16)	cis-1,2-Dichloroethene	(T)	11.06(.938)	1.74699	10.00
	Mass Abun				
	61.0 100.00	Quant Ion:	96.0	Ref Namr: TDCE12	
	96.0 66.00				
	98.0 41.00				
	RT Window: <None>	Peaks/min: <None>			
	Minimum Area: <None>	Peak/Base error: <None>			
	Slope sens: <None>	Subtraction Method:			
	Ignore max:	Auto Qdel Method:			

Label Methods 1-5: N,R,S,#,U

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
17)	Chloroform	(T)	11.49(.975)	3.99303	10.00
	Mass Abun				
	83.0 100.00	Quant Ion:	83.0	Ref Namr: CHCL3	
	85.0 65.80				
	RT Window: <None>	Peaks/min: <None>			
	Minimum Area: <None>	Peak/Base error: <None>			
	Slope sens: <None>	Subtraction Method:			
	Ignore max:	Auto Qdel Method:			

Label Methods 1-5: N,R,S,#,U

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
18)	Bromochloromethane	(T)	11.35(.963)	.89470	10.00
	Mass Abun				
	49.0 100.00	Quant Ion:	128.0	Ref Namr: BCM	
	130.0 60.00				
	128.0 47.00				
	RT Window: <None>	Peaks/min: <None>			
	Minimum Area: <None>	Peak/Base error: <None>			
	Slope sens: <None>	Subtraction Method:			
	Ignore max:	Auto Qdel Method:			

Label Methods 1-5: N,R,S,#,U

Comp No.	Compound Name	(Type)	Retention Time Min. (Rel)	R.F.	Conc.
19)	1,1,1-Trichloroethane	(T)	11.68(.992)	3.71881	10.00
	Mass Abun				
	97.0 100.00	Quant Ion:	97.0	Ref Namr: TCE111	
	99.0 63.00				
	41.0 57.00				